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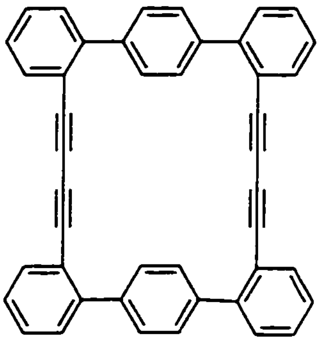
LIST OF ABBREVIATIONS

Δ	Reflux temperature
Bu	Butyl
Cp	Cyclopentadienyl
dba	Dibenzylidene acetone
dppm	Diphenylphosphinomethane
DEA	Diethylamine
DIPA	Diisopropylamine
DMAD	Dimethylacetylene Dicarboxylate
DME	Dimethoxyethane
DMF	Dimethylformamide
DMP	Dess Martin Periodinane
Dec	Decyl
DBA	Dehydrobenzoannulene
FVP	Flash Vacuum Pyrolysis
HEB	Hexaethynylbenzene
LDA	Lithium Diisopropylamide
LiHMDS	Lithium Hexmethyldisilylazide
Mes	Mesityl
NBS	*N*-bromosuccimide
NIS	*N*-iodosuccimide
NMP	*N*-methylpyrrolidine
ODCB	*o*-Dichlorobenzene
py	Pyridine
rt	Room Temperature
TBAF	Tetrabutylammonium fluoride
TBS	*t*-Butyldimethylsilyl
TEA	Triethylamine
TEM	Transmission Electron Microscopy
Tf	Trifluoromethylsulfonyl
TFA	Trifluoromethylsulfonic acid
THF	Tetrahydrofuran
TLC	Thin Layer Chromatography
TIPS	Triisopropylsilyl
TIPSA	Triisopropylsilylacetylene
TMEDA	Tetramethylethylenediamine
TMS	Trimethylsilyl
TMSA	Trimethylsilylacetylene
tol	Toluene

ABSTRACT

The synthesis of a novel family of acetylenic cyclophanes *via* Pd, Cu and Zn catalyzed cross-coupling reactions is described. The cyclophanes were constructed in good yields and X-ray crystal analysis revealed a twisted helical geometry. The nature of the geometry and the ability to complex solvent molecules within the lattice varied upon the number of acetylenic linkages present. Functionalized cyclophanes bearing long alkyl chains based on the helical structures **191** and **213** were also synthesized with potential as novel liquid crystals. Cyclophane **280** showed LC-like behavior when melting was observed under a polarized light microscope. Intramolecular cyclization of paracyclophanes was observed and resulted in cyclophanes, **192** and **203**. X-ray crystallographic analysis of the carboxylic acid derivative, **203** revealed the strained nature of the butadiyne bridge. The triple bonds were distorted with bond angles of 163.7° and 163.5°. A novel method for the synthesis of diynes and tetraynes using an *in situ* desilylation/dimerization procedure was developed but was unsuccessful in producing linear hexaynes. Attempted dimerization of **281** for the synthesis of **282** failed due to a competing intramolecular producing **283**. Derivative **307** revealed another highly strained butadiyne bridge possessing bond angles of 164.1° and 153.4°. Metacyclophanes with a termini separation of approximately between 7.8 and 10 Å did not undergo intramolecular cyclization. A sequential coupling procedure involving a double dimerization of acetylenes produced traces of the desired cyclophane **282** and no competing intramolecular cyclization products were observed. Progress towards **327**, a structural isomer of **282**, using an alternative sequential coupling protocol involving a double Sonogashira cyclization is detailed. Attempts to prepare acetylenic cyclophanes bearing metaphenyl/acetylene linkages are described. Initial investigations into the incorporation of thiophenes to acetylenic cyclophanes were thwarted by the instability of the precursor, **372**.



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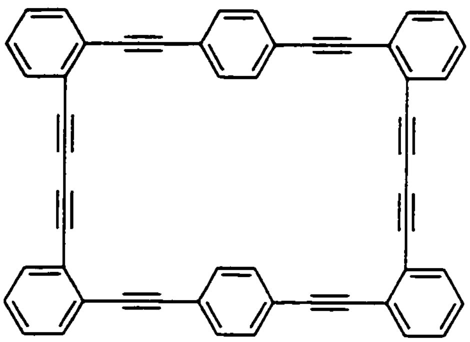

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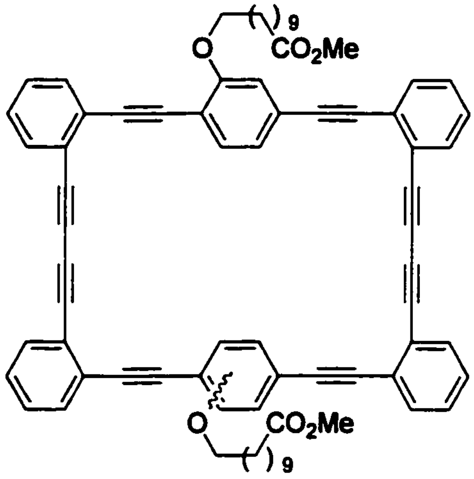

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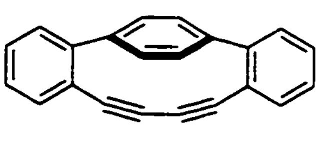

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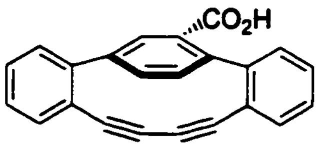

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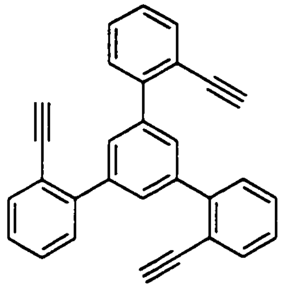

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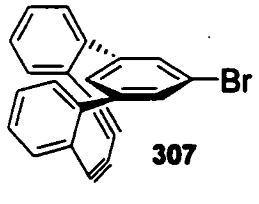

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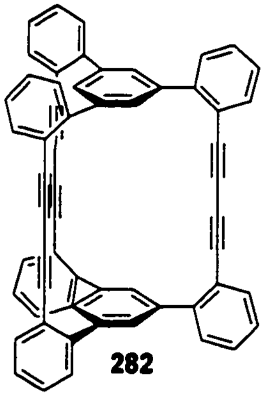

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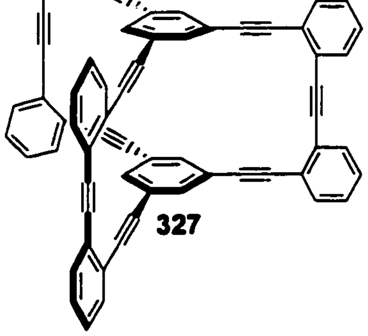

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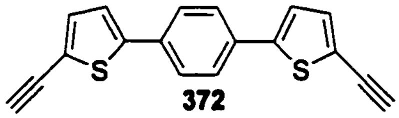

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ACKNOWLEDGEMENTS

First, I'd like to thank my supervisor, Dr. Fallis for his constant encouragement and excitement about chemistry. Most importantly, I've always felt that there was more to being a chemist than figuring out reaction schemes and mechanism- that there was so much you couldn't learn from a textbook. These could include learning the best attitudes about chemistry, different philosophies concerning science in general, about publishing, teaching, presentations and communicating your own ideas, the differences between academia and industry...the list could go on and on. All of these things required someone to take the time, a lot of time, to show and teach. Thank you for taking so much time to teach about all these things and more.

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I can't put into words all that you've done to make this a reality.
It could have never been done without your love.

CHAPTER 1: INTRODUCTION

1.1 Chemistry of the Carbon-Carbon Triple Bond.

The carbon – carbon triple bond is one of the simplest functional groups in organic chemistry. Despite its relative simplicity, it remains actively studied and its chemistry continues to attract the interest and imagination of synthetic chemists. Acetylene chemistry has experienced perhaps its most active exploration since the 1950's and 1960's. In the mid 1980's, the carbon-carbon triple bond had again become a prevalent functionality in the field of chemistry. A large family of potent antitumour antibiotics containing *cis*-enediyne fragments had emerged as challenging synthetic targets. The actived triple bond units of these promising natural products were capable of undergoing Bergman cyclization, enabling proton abstraction and subsequent cleaving of DNA strands.

Perhaps the most extensive usage of carbon-carbon triple bonds, however, has come in the field of material science. Acetylene units have become the major structural unit in the synthesis of molecular scaffolds. Acetylenes are involved in the construction of nanosized molecular objects with discrete 3-dimensional structures. Their incorporation into existing carbon frameworks has led to new variations and modifications of the carbon allotropes of graphite and diamond. They have proved instrumental in the ongoing efforts to develop a laboratory synthesis of fullerenes and their endohedral cousins. They have been inserted into the backbone of polymers leading to novel scaffolds. These new molecules have demonstrated unprecedented electrical, structural and optical properties.

New nanosized molecular targets continue to be constructed with a variety of architectures, shapes and geometries. The sole limitation to the synthesis of these targets seems to be the development of the appropriate building blocks. The geometry and ease of functionalization of the acetylene unit allows the molecular scaffolding to be "shape persistent". The nanosized targets provide a large molecular area and surface, and in combination with the ability to maintain a discrete 3-dimensional structure, allow for the construction of even larger arrays through supramolecular interactions. These molecules are often referred to as nanostructures, although the term is used frequently to describe such structures without an established rigid definition. The obvious geometry of the acetylene unit has the possibility to lead to the construction of a variety of non-natural molecular systems. These shape persistent features have allowed for the rational design of molecular crystals, liquid crystals, monolayer surfaces, and novel 2-dimensional and 3-dimensional allotropes of carbon.

The synthesis of these novel targets has coincided with the constant development of new synthetic methods for the construction of carbon-carbon bonds. Various cross-coupling reactions for the connection of Csp-Csp, Csp-Csp^2 and Csp^2-Csp^2 centers have been investigated and optimized and these synthetic protocols remain crucial to the synthesis of molecular scaffolding. Furthermore, it has been demonstrated that molecules with a wide degree of structural diversity could be achieved using a small set of reactions. The development of novel synthetic methods and the construction of new materials within the past two decades have broadened the boundaries of synthesis. "Synthesis" includes not only small molecules, but the construction and design of macromolecules as well. It encompasses the formation of covalent bonds *and* the formation and organization of molecules based on weaker bonding interactions.

1.2 Methods For Carbon-Carbon Bond Formation: C_{aryl}-C_{aryl}, C_{sp}-C_{sp}, and C_{sp}-$C_{sp}{}^2$ Coupling Reactions.

1.2.1 $C_{sp}{}^2$-$C_{sp}{}^2$ Coupling Reactions.

Just over twenty years ago, Pd-catalyzed cross-coupling reactions were merely a scientific curiosity. Due to the high price of palladium, there was much skepticism about the synthetic usefulness of Pd-based methods. Today, despite the cost, Pd-catalyzed cross-couplings are recognized as the most general and widely used synthetic methods for carbon-carbon bond formation. Their versatility is displayed in the number and variety of organometallic partners it can react with, among them Mg, Zn, B, Al, Sn, Cu, and Zr.[1] Pd-catalyzed cross-couplings are now being developed as enantioselective processes, and as components of tandem and cascade reactions for application towards total syntheses of natural products, drugs, and towards material science and engineering.

---

[1] Negishi, E.; Liu, F. In *Metal-Catalyzed Cross-coupling Reactions;* Deiderich, F.; Stang, P.J., Eds.; Wiley-VCH, Weinheim, Germany, 1998; pp 1- 48.

Perhaps the most widely studied of the palladium based methodologies is the Stille coupling of organic halides with organotin derivatives (Figure 1).[2] Extensive mechanistic studies have been performed on the reaction and the roles of all its components.[3] These studies are often generalized to include reactions with other counterions or organometallic reagents. The basic catalytic cycle of the palladium based Stille coupling is outlined in Figure 1.

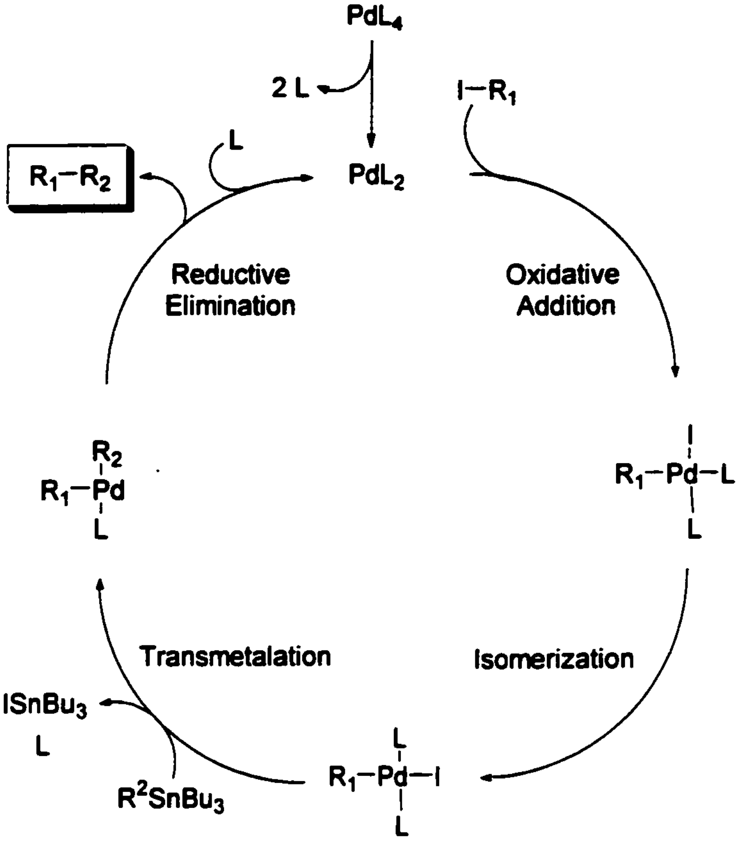

Figure 1. Basic mechanism of palladium catalyzed cross-coupling reactions.

The Stille coupling often utilizes Pd^0 based catalysts, which must lose two ligands to give the active 14 electron Pd species. Oxidative addition of the organic halide is followed by a fast isomerization to arrange the ligands for transmetalation.

---

[2] (a) Stille, J.K. *Angew. Chem. Int. Ed. Engl.* **1986**, *25*, 508. (b) McKean, D.R.; Parrinello, G.; Renaldo, A.F.; Sille, J.K. *J. Org. Chem.* **1987**, *52*, 422. (c) Scott, W.J.; Stille, J.K. *J. Am. Chem. Soc.* **1986**, *108*, 3033.

[3] For some early mechanistic investigations see (a) Farina, V.; Krihnan, B.; Marshall, D.R.; Roth, G.P. *J. Org. Chem.* **1993**, *58*, 5434. (b) Farina, V.; Kapadia, S.; Krishnan, B.; Wang, C.; Liebeskind, L.S. *J. Org. Chem.* **1994**, *59*, 5905. (c) Segelstein, B.E.; Butler, T.W.; Chenard, B.L. *J. Org. Chem.* **1995**, *60*, 12. (d) Allred, G.D.; Liebeskind, L.S. J. Am. Chem. Soc. **1996**, *118*, 2748. (e) Roth, G.P.; Farina, V. *Tetrahedron Lett.* **1995**, *36*, 2191.

Transmetallation occurs as the unsaturated moiety on the organotin derivative is exchanged for a halide in the palladium complex. A second ligand is displaced and the R groups are orientated *cis* to one another as required for reductive elimination. After the desired product is released from the complex, association of a ligand regenerates the active palladium catalyst.

Recent mechanistic studies by Espinet[4] have shown that the transmetalation step of the reaction may occur *via* the transition state depicted in Figure 2. This process leads to a T-shaped three co-ordinate *cis*-[PdR_1R_2L] species from which irreversible reductive elimination must be fast.

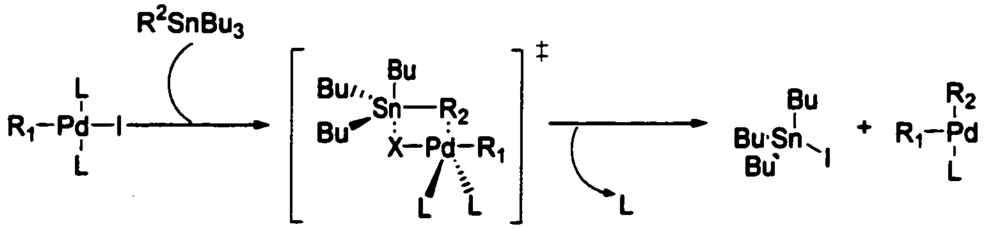

Figure 2. The transmetallation step of the Stille reaction.

1.2.2 C_{sp}-C_{sp}^2 Coupling Reactions: The Sonogashira Coupling.

During the past two decades, the significant advances in the field of transition metal mediated coupling reactions have generated new methods for the construction of C_{sp}-C_{sp}^2 bonds. These compounds are often synthesized by the use of two variations of a cross coupling reaction. The transition metal catalyzed formation of C_{sp}-C_{sp}^2 bonds can be achieved through coupling of alkynyl or alkynyl halides to metal aryls or by coupling of metal alkynyl reagents to organic aryl halides. Particularly popular, is the *Sonogashira coupling.*[5,6] It is a facile Pd-catalyzed cross coupling of aryl and alkenyl halides with terminal alkynes in the presence of a cocatalyst such as copper iodide in amine solvents. The simple and mild conditions required make it especially attractive. The proposed reaction mechanism is described in Figure 3.

The mechanism of the reaction is based on two cyclic processes. The overall reaction is catalytic; there is no acceleration in the reductive elimination step although co-

---

[4] Casado, A.L.; Espinet, P. *J. Am. Chem. Soc.* **1998**, *120*, 8978.

[5] Sonogashira, K.; Tohda, Y.; Higihara, N. *Tetrahedron Lett.* **1975**, *16*, 4467.

[6] Sonogashira K. In *Metal-catalyzed Cross-coupling Reactions;* Deiderich, F.; Stang, P.J., Eds.; Wiley-VCH, Weinheim, Germany, 1998; pp 203- 227.

ordination of a copper ion to the alkynyl group is expected. As a source of palladium, $Pd(PPh_3)_2Cl_2$ is the common reagent. This reagent undergoes a homocoupling of two terminal acetylenes and generates the active 14 electron complex $Pd^0(PPh_3)_2$. Formation of an active copper acetylide is proposed in amine solvents, and can undergo transmetalation twice to the palladium catalyst. Reductive elimination would then give the required oxidation state for palladium. Small amounts of the dimer of the terminal acetylene are often observed in these reactions. Other palladium sources can be used, although $Pd(PPh_3)_4$ must lose two equivalents of PPh_3 and the reaction can be hampered by excess phosphine ligand. Addition of two equivalents of triphenylphosphine to $Pd(OAc)_2$, $Pd_2(dba)_3$, or $Pd(CH_3CN)_2Cl_2$ followed by reductive elimination also generates the active species.

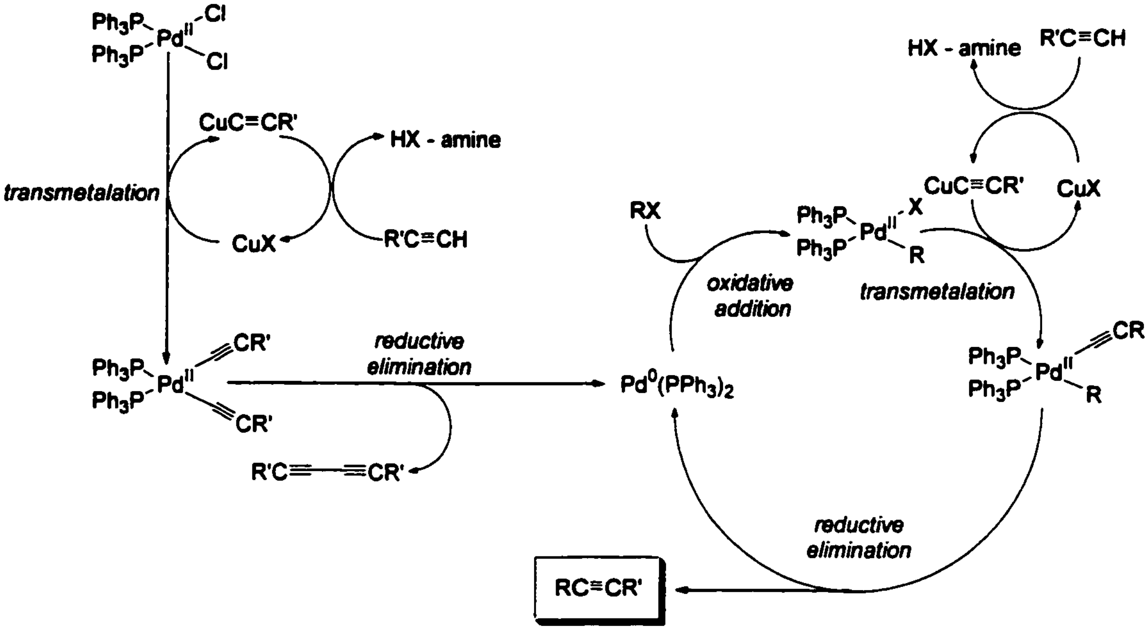

Figure 3. Palladium catalyzed cross-coupling reaction of terminal acetylenes with sp^2 halides.

The following step involves oxidative addition of the organic halide to the 14 electron species. Various organic halides can undergo reaction and the order of reactivity has been established as vinyl iodide ≅ vinyl bromide > aryl iodide > vinyl chloride >> aryl bromide. Vinyl iodides, bromides and chlorides and aryl iodides are all reactive enough to undergo reaction at room temperature. For aryl bromides, higher reaction temperatures are often required, although novel phosphine ligands have been developed to overcome this synthetic difficulty.[7] Little to no reaction is observed without the addition of CuI as a cocatalyst. Transmetalation of another equivalent of the copper acetylide followed by reductive elimination releases the cross-coupled product and regenerates the active palladium species. The role of the amine base is extremely important. Diethylamine and triethylamine, often with additional THF as a cosolvent, remain the solvent system of choice. Various other amine bases can be used and particularily when organic triflates are used as coupling partners, may give overall better yields and rates than the traditional bases.

1.2.3 C_{sp}-C_{sp} Coupling Reactions: Cu-Mediated Oxidative Acetylenic Coupling.

Di- and oligoacetylene moieties are frequently encountered in natural products. Their interesting electronic and optical properties have spurred research into new linear and cross-linked oligoalkynes and carbon allotropes. These synthetic applications and challenges have in turn spawned new methods and protocols for their formation. Despite the variety of acetylenic coupling protocols, Cu-mediated oxidative coupling remains the method of choice among organic chemists. Although there have been many synthetic variations and adaptations of the classic Glaser,[8] Eglinton,[9] and Hay couplings,[10] the mechanism is not understood.[11]

Studies have shown that the mechanism is highly dependent on the experimental set-up, making it difficult to compare the results of various kinetic investigations.[12] The earliest proposals concerned the recombination of acetylenic radicals to give dimerization products. After several early kinetic investigations proved that in alkaline media, copper ions serve as direct oxidizing agents, Zalkind and Fundyler proposed a heterolytic cleavage and single electron transfer to generate the proposed radical intermediates (Figure 4).[12]

---

[7] Hundertmark, T.; Littke, A.F.; Buchwald, S.L.; Fu, G.C. *Org. Lett.* **2000**, *2*, 1729 and reference cited therein.

[8] (a) Glaser, C. *Ber. Dtsch. Chem. Ges.* **1869**, *2*, 422. (b) Glaser, C. *Ann. Chem. Pharm.* **1870**, *154*, 137.

[9] Eglinton, G.; Galbraith, A.R. *Chem. Ind. (London)* **1956**, 737.

[10] Hay, A.S. *J. Org. Chem.* **1962**, *27*, 3320.

[11] For an excellent review and discussion of acetylenic coupling see Siemsen, P.; Livingston, R.C.; Diederich, F. *Angew. Chem. Int. Ed. Engl.* **2000**, *39*, 2632.

[12] (a) Zalkind, Y.S.; Fundyler, Fr.B. *Ber. Dtsch. Chem.* **1936**, *69*, 128. (b) Zalkind, Y.S.; Fundyler, Fr.B. *J. Gen. Chem. USSR* **1957**, *27*, 3008.

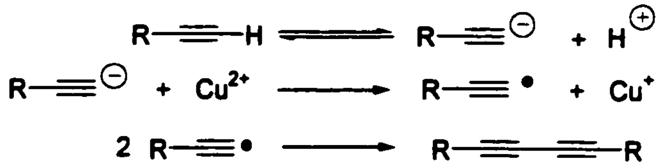

Figure 4. Early proposal by Zalkind and Fundyler for the mechanism of oxidative acetylenic coupling.

Bohlmann questioned the existence of free radical intermediates. He noted that mixtures of electronically different alkynes gave predominantly the homocoupled products. No such selectivity should be observed with free radicals. The study on rates of dimerization showed that more acidic acetylenes undergo more rapid dimerization under alkaline conditions. These observations coincided with and reinforced the experimental evidence of several other research groups concerning the role of Cu(I) and Cu(II) ions in the reaction. It was proposed that complexation of Cu(I) ions to the triple bond activated it towards deprotonation.

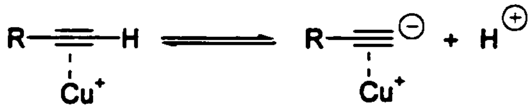

Figure 5. Bohlmann's proposed activation of acetylenes through π-complexation.

This explains the trend observed that more conjugated substrates react more slowly.[13] A multitude of questions exist for the exact nature of the complexation of copper ions to triple bonds, including which oxidation states are involved, whether there exists non-reactive π-complexes and whether more than a single copper ion are necessary. In addition, various studies have also been performed to examine the role of Cu(I) ions in buffered and non-buffered reaction solutions, in the absence or presence of oxygen, and with additional background electrolytes. Despite the collection of experimental data to date, no universal mechanistic explanation has been put forth for the coupling.

The mechanism provided by Bohlmann 36 years ago, is still accepted as the most reasonable picture. The observed second order rate dependence on alkyne concentration resulted in a proposed dimeric or dinuclear copper(II) acetylide complex (Figure 6). This

---

[13] Bohlmann, F.; Schonowsky, H.; Inhoffen, E.; Grau, G. *Chem. Ber.* **1964**, *97*, 794.

mechanism consists of simultaneous oxidation and reductive elimination and C-C bond formation to give products. Dioxygen or Cu(I) ions may serve as co-oxidants to regenerate the functional Cu(II) ions depending on the experimental conditions.

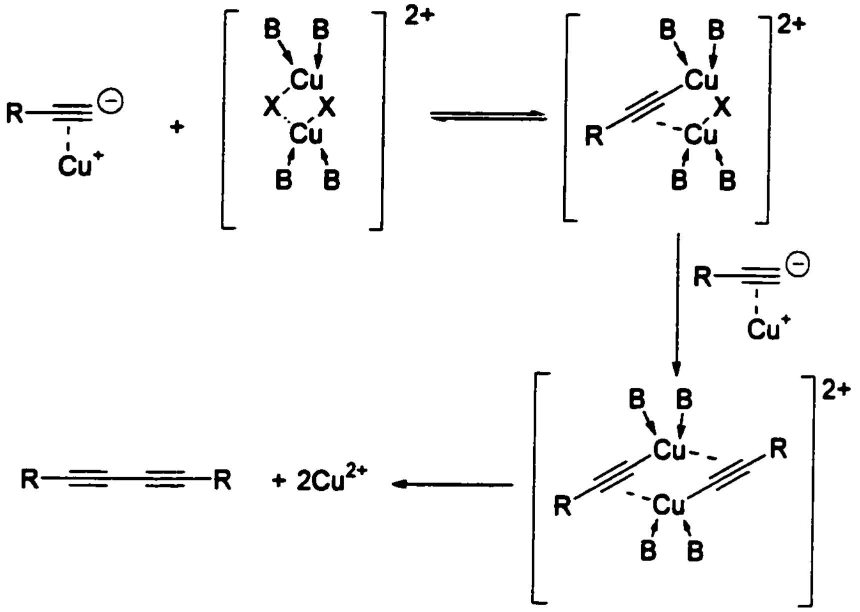

Figure 6. Dimeric copper acetylides first proposed by Bohlmann et. al. as intermediates in oxidative acetylenic coupling. B = N ligand (pyridine) and X = Cl⁻, OAc.

1.3. Polyethynylated Carbon Networks

Carbon rich materials with novel shapes and topologies are of interest due to their possible tunable electronic and optical properties.[14] Carbon networks with delocalized π-systems are particularly promising due to the changes in electronic properties that can be effected by altering their geometric characteristics and environment. Accordingly, extended π-systems have been modified and applied to various carbon 2- and 3-dimensional networks. The simplest carbon networks, graphite and diamond, can be modified to form polyethynylated π-systems by replacement of single C-C bonds by acetylene or 1,3-butadiyne units.[15] Consequently, individual units or portions of these infinite networks can be synthesized and investigated as models of these novel materials. These "bits and pieces" are often acetylenic cyclophanes of varying 3-dimensional

---

[14] (a) *Topics in Current Chemistry (Carbon Rich Compounds* I); de Meijere, A., Ed.; Springer: Berlin, 1998; Vol. 196. (b) *Topics in Current Chemistry (Carbon-Rich Compounds II)*; de Meijere, A., Ed.,; Springer: Berlin, 1999; Vol. 201.
[15] Bunz, U.H.F.; Rubin, Y.; Tobe, Y. *Chem. Soc. Rev.* **1999**, *28*, 107.

geometries. Planar cyclophanes represent models for 2-dimensional system while other acetylenic cyclophanes having helical twisted conformations may give access to carbon nanotubes. Cyclophanes[16] connected by acetylenic bridges form rigid cage-like molecules. In many cases, the flexibility of the carbon-carbon triple bond allows these molecules to bend and adopt interesting strained conformations.

1.3.1 Planar or 2-Dimensional Carbon Networks

There has been an increase in the study of dehydrobenzoannulenes (DBAs) and dehydroannulenes as these molecules were proposed as possible precursors to carbon networks.[17] These molecules were previously difficult to obtain *via* traditional Cu-mediated dimerizations/cyclooligomerizations of α,ω-diacetylenes. The original method produced complex mixtures of products making isolation and purification problematic. Recently, these systems have also been systemically constructed to study their various electronic and optical properties. This systematic synthesis was based on an intramolecular ring closure strategy and has allowed the preparation of several novel DBAs containing an odd number of acetylene linkages (Scheme 1).[18,19a]

Haley and co-workers have also built a series of "alkyne-interrupted" DBAs where one or more of the connecting alkyne units has been replaced by an olefin. Their study has shown that highly alkynylated dehydrobenzoannulenes do indeed possess ring currents, *albeit* weakly so. They were unable to quantify the ring current.

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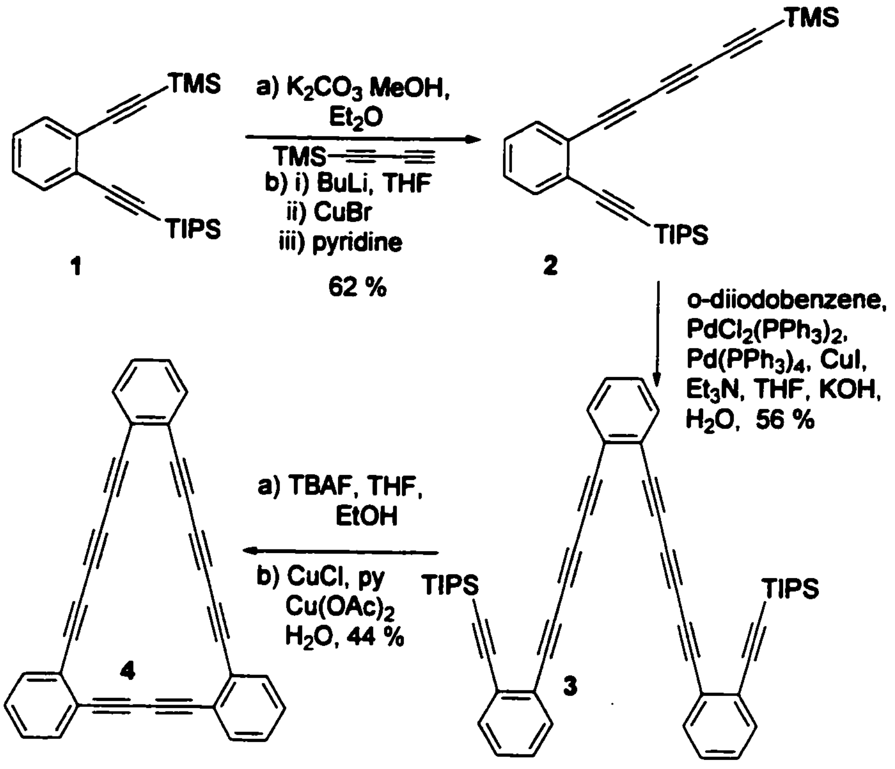
[16] (a) *Top. Curr. Chem.*;1983, 113. (b) *Top. Curr. Chem.*;1983, 115. (c) *Cyclophanes*, Keehn, P. M., Rosenfeld S. M., Eds.; Academic Press, New York, 1983, Vols I, II. (d) Diederich, F. *Cyclophanes*; Royal Society of Chemistry, Cambridge, UK, 1991. (e) Vogtle, F. *Cyclophane Chemistry*; Wiley, New York, 1993. (f) *Top. Curr. Chem.*; 1994, 172. (g) Bodwell, G. J. *Angew. Chem. Int. Ed. Engl.* **1996**, *35*, 2085. (h) de Meijere, A.; Konig, B. *Synlett* **1997**, 1221.

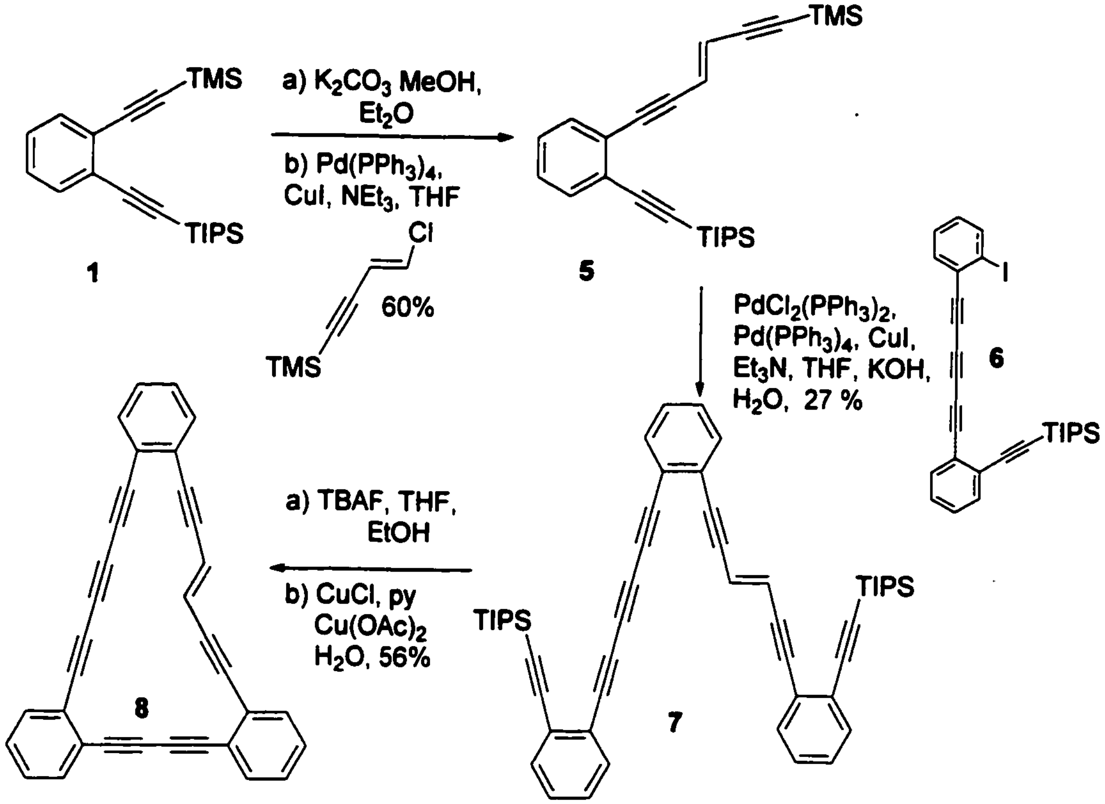
[17] Haley, M.M. *Synlett* **1998**, 557.

[18] (a) Haley, M.M.; Bell, M.L.; English, J.J.; Johnson, C.A.; Weakley, T.J.R. *J. Am. Chem. Soc.* **1997**, *119*, 2956 (b) Haley, M.M.; Wan, W.B. *Advances in Strained and Interesting Organic Molecules*; Halton, B., Ed.; JAI Press: New York, 2000; Vol. 8, pp 1-41.

[19] (a) Wan, W.B.; Kimball, D.B.; Haley, M.M. *Tetrahedron Lett.* **1998**, *39*, 6795. (b) Matzger, A.J.; Vollhardt, K.P.C. *Tetrahedron Lett.* **1998**, *39*, 6791.

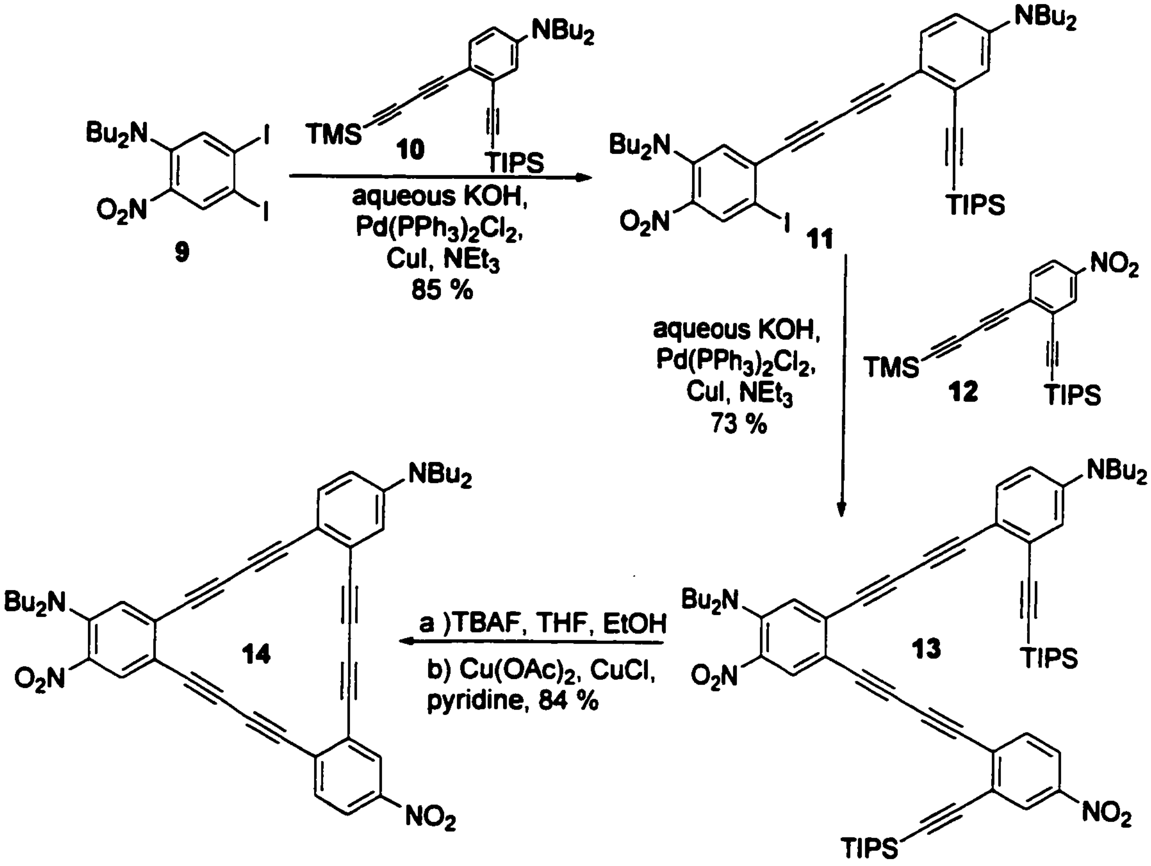


Scheme 1. Preparation of novel DBA's containing 1,3,5-hexatriyne linkages.



Scheme 2. Synthesis of "alkyne-interupted" DBA's for the study of induced ring currents and aromaticity.

Haley extended the technology to generate highly functionalized dehydro[18]benzoannulenes. The functionality is incorporated at the building block level and therefore enables one to have complete control over the ring substitution pattern.[20] This synthetic strategy generated a series of substituted [18]-DBAs and permitted the first structure-property relationship (SPR) studies of these molecules. It was determined that these compounds exhibit large bathochromic shifts as stronger donor or acceptor functionalities were incorporated. The authors are currently investigating the optical properties of these molecules and their thermal polymerization process.



Scheme 3. Synthesis of highly substituted dehydro[18]benzoannulenes.

While dehydrobenzoannulenes find application as ligands for organometallic chemistry, as hosts for binding guest molecules, and as probes for induced weak ring currents, their most useful application is towards carbon rich materials. Although perethynylated compounds have long been considered prime candidates as precursors and mimics for carbon rich materials, they posses several disadvantages. Contrary to

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[20] Pak, J.J.; Weakley, T.J.R.; Haley, M.M. *J. Am. Chem. Soc.* **1999**, *121*, 8182.

perethynylated compounds, DBAs tend to be stable to heat, light, and oxygen and should serve as superior potential macrocyclic mimics of carbon networks.

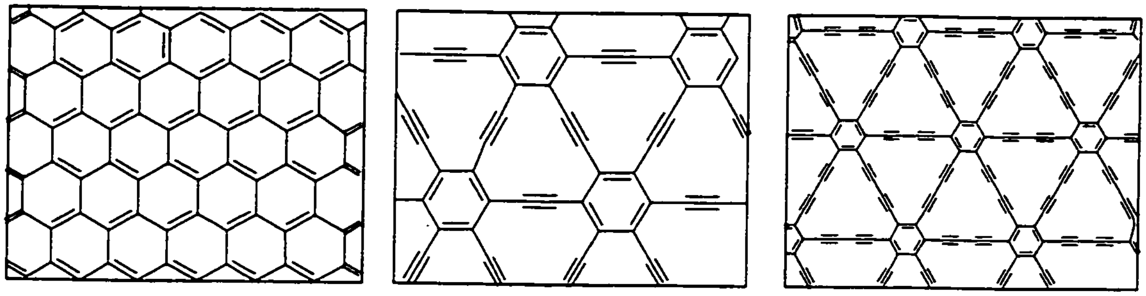
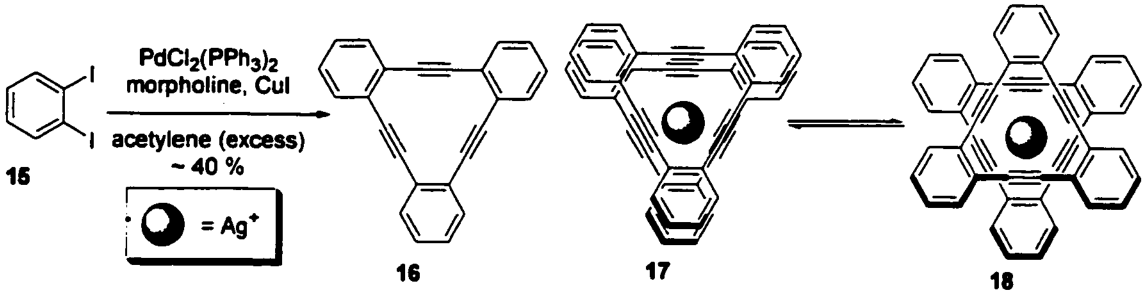


Figure 7. Several modified 2-dimensional carbon allotropes of graphite (left): graphyne, where all single bonds have been replaced by an ethyne unit (center), and graphdiyne, where the single bonds have been replaced by 1,3-butadiyne linkages.

The carbon-carbon allotrope "graphyne" has recently been proposed as a stable form of carbon shown in Figure 7.[21] It is predicted to have strong nonlinear optical behavior, be a large band gap semiconductor (E_g = 1.2 eV) and its alkali metal charge complexes are expected to be metallic in nature.



Scheme 4. Small [12]annulenes are capable of binding silver ions and may serve as mimics of the graphyne network.

Iyoda and co-workers have synthesized a small portion of the graphyne network in the form of simple [12]annulenes. The small [12]annulene, **16**, isolated after exposing 1,2-diiodobenzene and a palladium catalyst to excess acetylene gas, may serve as a mimic of graphyne and its complexing abilities.[22] These interesting [12]annulenes have been found to form stable $AgSO_3CF_3$ complexes in solution and NMR studies have shown that they exist predominately in the eclipsed form, **17** (Scheme 4).

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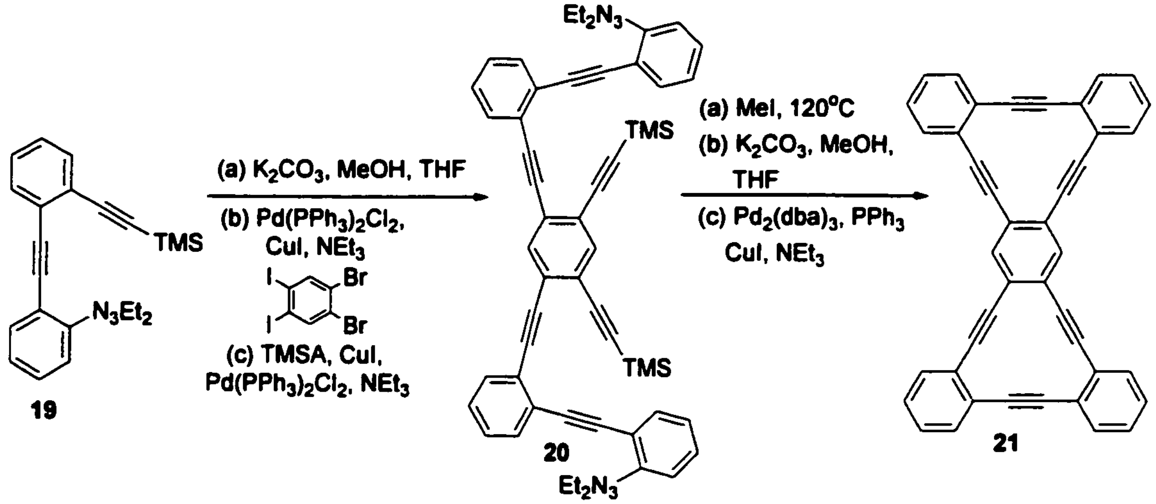
[21] (a) Baughman, R.H.; Eckhardt, H.; Kertesz, M.J. *J. Chem. Phys.* **1987**, *87*, 6687. (b) Narita, N.; Nagagi, S.; Suzuki, S.; Nakao, K. *Phys. Rev. B.* **1998**, *58*, 11000 (c) Youngs, W.J.; Tessier, C.A.; Bradshaw, J.D. *Chem. Rev.* **1999**, *99*, 3153.

[22] Iyoda, M.; Vorasingha, A.; Kuwatani, Y.; Yoshida, M. *Tetrahedron Lett.* **1998**, *39*, 4701.

Synthetic accessibility has been the major deterrent in the efforts to study graphyne. Haley and co-workers have demonstrated that phenyl-acetylene constructs can serve as network mimics. A macrocyclic segment has been synthesized using the intramolecular ring closure strategy and is outlined in Scheme 5.[23]



Scheme 5. Macrocyclic segments of the graphyne network synthesized by an intramolecular ring closure strategy

The protocol has been applied towards mimics of the extended grapydiyne structure (Figure 7). Graphdiyne has been calculated to be the most stable allotrope of carbon bearing butadiyne bridges or linkages.[24] It is expected to exhibit similar properties as graphyne, although it may display enhanced REDOX properties. Graphydiyne has large holes or cavities approximately 2.5 Å in diameter within its planar sheets that may facilitate through-sheet transport of small ions and may accommodate a variety of larger metal ions. The novel carbon allotrope[25] may also have intersheet dopant properties not available to graphite. Haley's synthesis of a macrocyclic segment is described in Scheme 6. Analysis of **24** revealed a global aromaticity and ring current contradictory to existing theories of extended DBAs.

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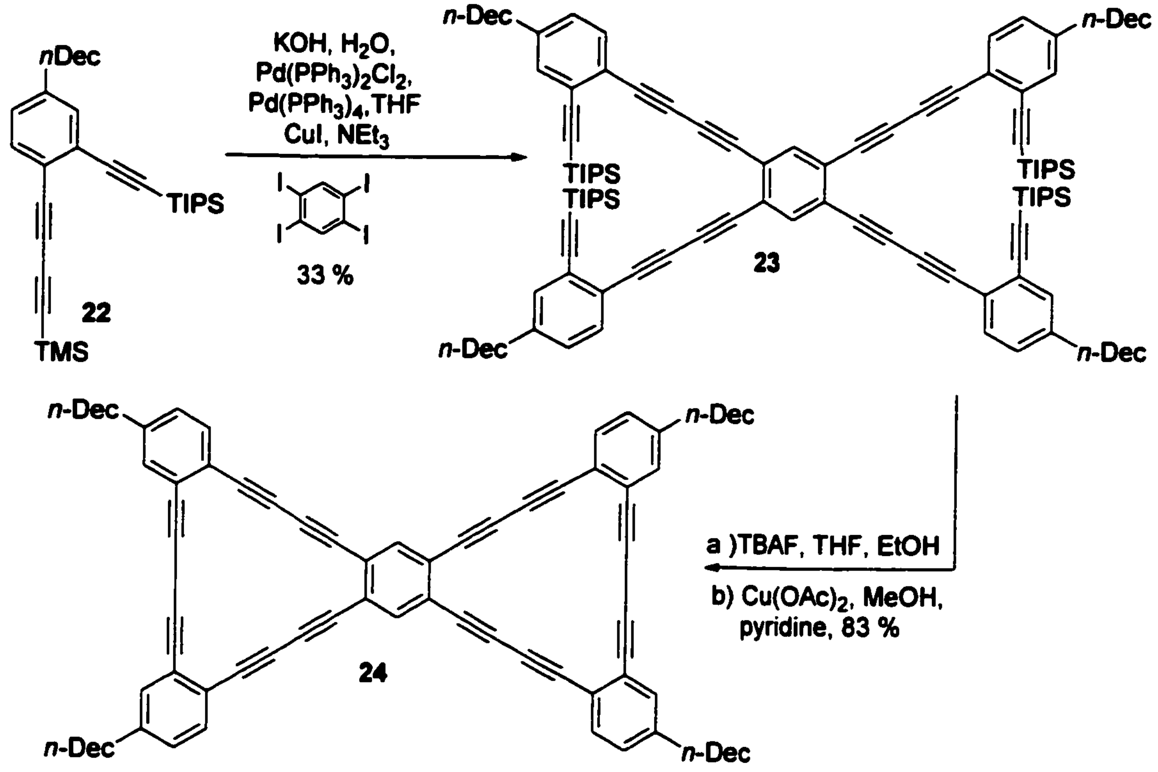
[23] Kehoe, J.M.; Kiley, J.H.; English, J.J.; Johnson, C.A.; Peterson, R.C.; Haley, M.M. *Org. Lett.* **2000**, *2*, 969.

[24] Haley, M.M.; Brand, S.C.; Pak, J.J. *Angew. Chem. Int. Ed. Engl.* **1997**, *36*, 836.

[25] Although allotrope refers to isomers of elements, this term has also been used to describe *graphyne* and *graphdiyne*.



Scheme 6. Synthesis of "alkyne-interupted" DBA's for the study of induced ring currents and aromaticity.

Although much progress has been made towards the synthesis of these carbon allotropes *via* dehydrobenzoannulenes structures, there is still much interest in constructing these compounds through the use of hexaethynylbenzenes (HEBs). Rubin has prepared 1,3,5/2,4,6 differentially substituted benzenes (Scheme 7). The electron rich systems display interesting fluorescence properties and the potential to lead to novel discotic liquid crystals.[26]

Another synthetic approach to building substituted hexaethynylbenzenes of C_{2v} symmetry was the use of Diels-Alder reactions of tetraethynylcyclopentadienones. Tobe and Rubin independently studied the same synthetic route to this class of compounds. Tobe synthesized the tetraethynylcyclopentadiene derivative, **34**, by a Pd-catalyzed cross coupling of various tetrahalocyclopentadienone acetals (Scheme 8).[27]

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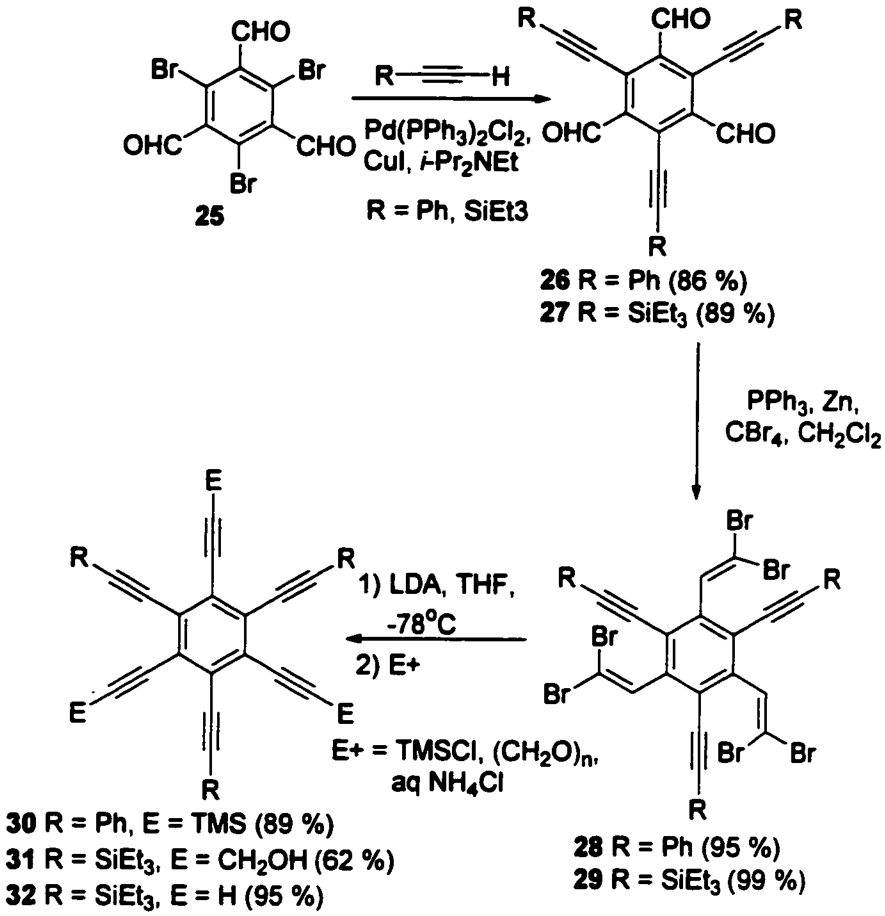
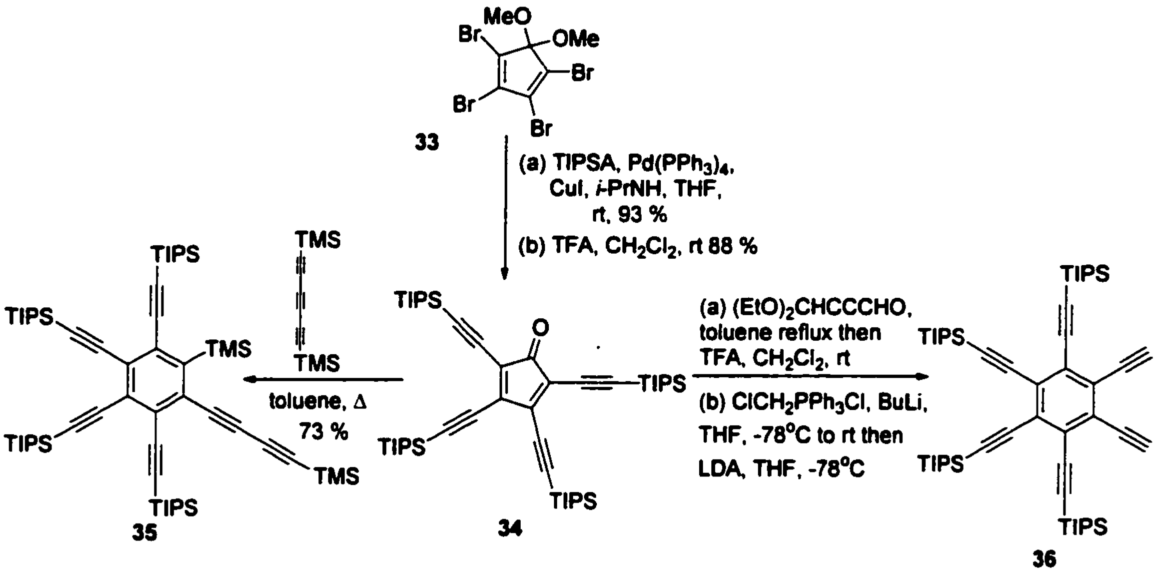
[26] Anthony, J.E.; Khan, S.I.; Rubin, Y. *Tetrahedron Lett.* **1997**, *38*, 3499.

[27] Tobe, Y.; Kubota, K.; Naemura, K. *J. Org. Chem.* **1997**, *62*, 3430.

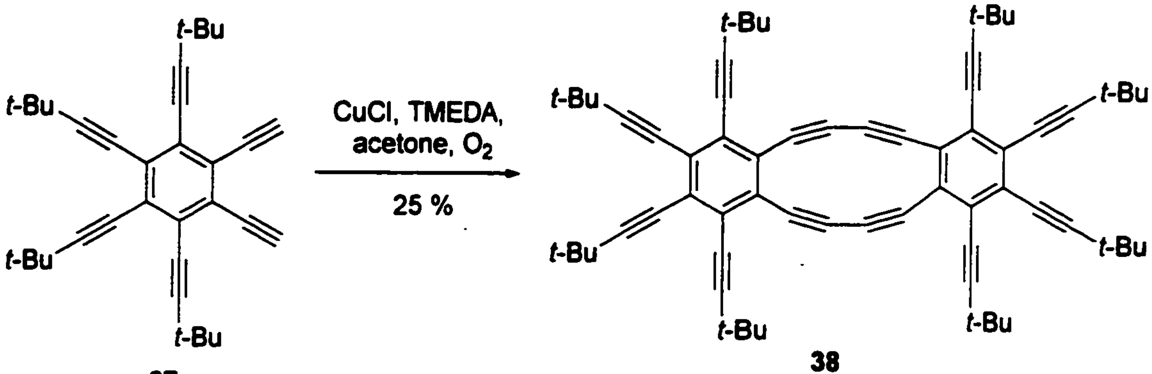


Scheme 7. Hexaethynylbenzenes with the potential to lead to discotic liquid crystals.



Scheme 8. Synthesis of hexaethynylbenzenes through Diel-Alder reaction of tetraethynylcyclopentadienones.

The cyclopentadienone, **34** undergoes a Diels-Alder reaction with 1,6-Di-(trimethylsilyl)hexa-1,3,5-triyne at room temperature to give **35** as the sole isomer. Similarly, by altering the choice of dienophile, the hexaethynylbenzene **36** could be constructed in good yield. Rubin also studied the Diels Alder reaction of various dienophiles with tetraethynylcyclopentadienones similar to **34**.[28] Again, when a Diels-Alder reaction with hexatriynes were attempted, the sole adduct isolated was the regioisomer where reaction had occurred at one of the terminal acetylenes. The steric encumbrance of the terminal trialkylsilyl groups would suggest reaction at the central alkyne. Interestingly, Rubin was able to synthesize the octade-hydro[12]annulene, **38**, as a stable yellow crystalline solid. The dimer exhibited strange bond elongations in the benzene ring and its stability was attributed to the ability of the large *t*-Bu groups and the $CHCl_3$ molecules present in the crystal packing structure to shield the sensitive [12] annulene core (Scheme 9).



37 **38**

Scheme 9. Synthesis of "alkyne-interupted" DBA's for the study of induced ring currents and aromaticity.

Several research groups have now developed protocols for the synthesis of hexaethynylbenzene from Pd-catalyzed cross coupling reactions. Tobe and co-workers have developed tandem Sonogashira/Negishi cross-coupling reactions for this purpose.[29] Ethynylzinc cross-coupling reagents have the strong ability to react with substrates typically difficult to prepare by normal Sonogashira methods. The strategy relies on the fact that chloroiodobenzenes exhibit different reactivities. After the iodides have reacted under normal Sonogashira reaction conditions, the weakly electron-withdrawing ethynyl

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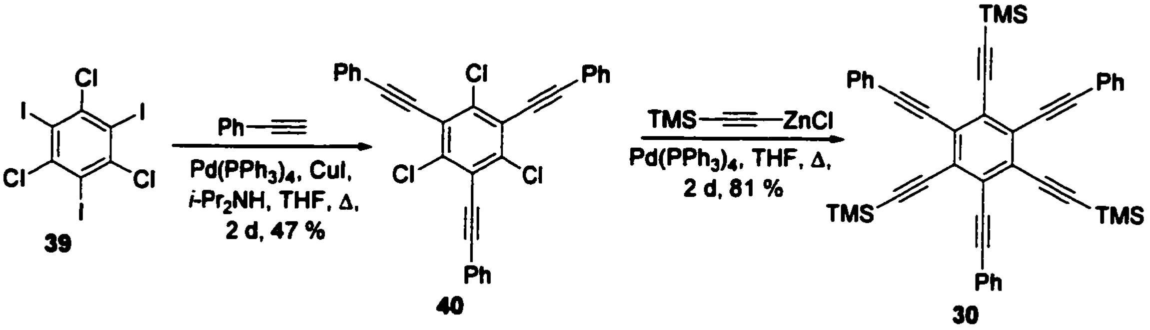
[28] Tovar, J.D.; Jux, N.; Jarrosson, T.; Khan, S.I.; Rubin, Y. *J. Org. Chem.* **1997**, *62*, 3432.

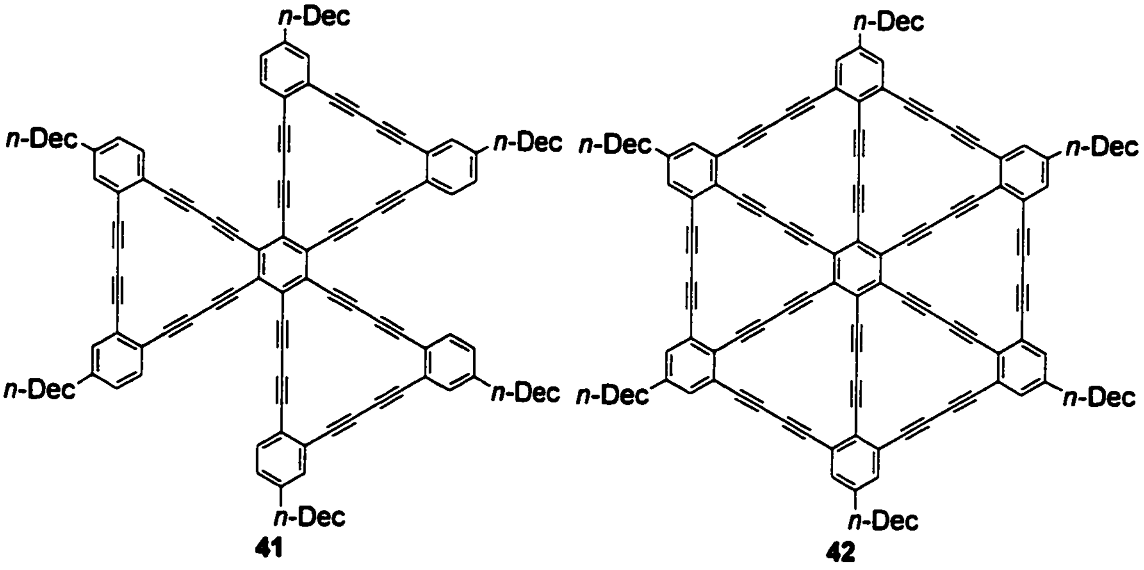
[29] Sonoda, M.; Inaba, A.; Itahashi, K.; Tobe, Y. *Org. Lett.* **2001**, *3*, 2419.

groups help to activate the chlorides towards reaction with the ethynylzinc reagents (Scheme 10).



Scheme 10. Synthesis of hexaethynylbenzene using a tandem Sonogashira/ Negishi coupling protocol.

Haley and Wan have also explored the six-fold cross-coupling of hexaiodobenzene using novel catalytic system for the generation of mimics of the carbon allotrope graphdiyne.[30] They focused their studies on the macrocyclic trefoil segment, **41** and the wheel unit, **42**. These units represent the largest segments of the graphdiyne allotrope synthesized to date.



Scheme 11. Trefoil and wheel shaped segments of the graphdiyne network.

Haley's success in six-fold cross-coupling of sensitive butadiynyl functionalities to hexaiodobenzene lies in the use of a modified Pd[P(*o*-Tol)$_3$]$_2$ catalyst. The large steric bulk of the ligands increases the cone angle from 145° (for PPh$_3$) to 195°. The larger

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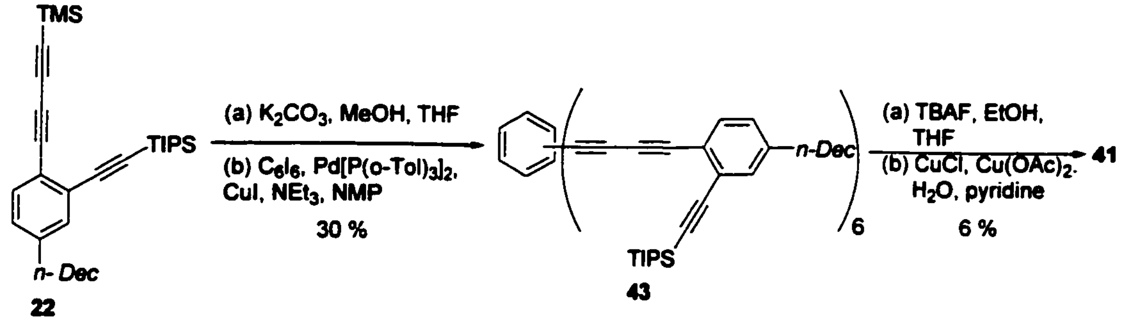
[30] Wan, W.B.; Haley, M.M. *J. Org. Chem.* **2001**, *66*, 3893.

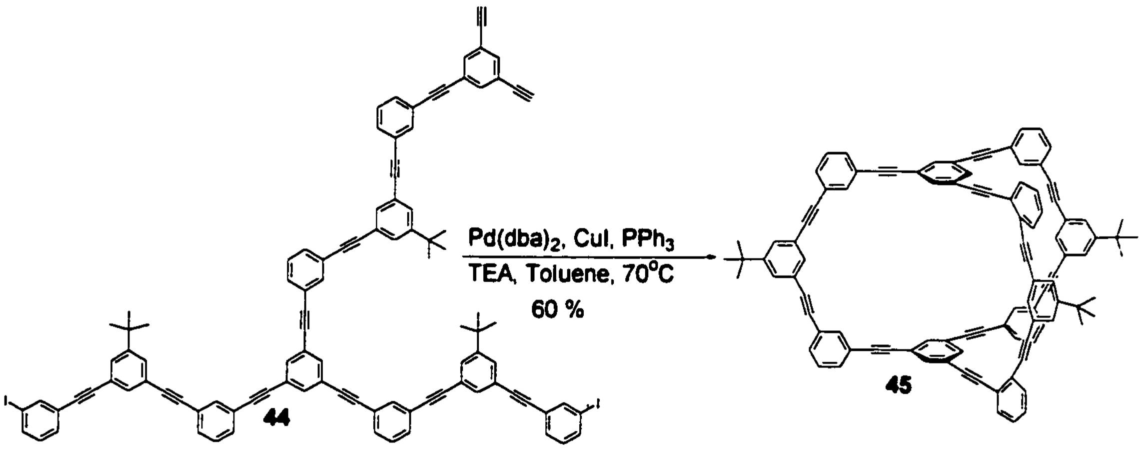
ligands readily dissociate creating a "naked" and reactive metal center without steric bulk, ideal for the coupling of bulky iodoarene species.



Scheme 12. Six-fold coupling of sensitive butadiynyl units to hexaiodobenzene.

1.3.2 3-Dimensional Carbon Networks

Phenyl acetylene nanostructures have proved to be attractive structures for novel molecular crystals, liquid crystals and monolayer surfaces. The flexibility of the geometry of phenyl acetylene linkages ranging from *ortho*, *meta*, or *para* allows for the very wide range of 3 dimensional structures to be created.[31] Moore and co-workers have used a site specific double cyclization of branched phenylacetylene oligomers to construct the discrete macrocycle, **45** (Scheme 13).[32]



Scheme 13. The double cyclization of Moore and co-workers allows for the construction of 3-dimensional macrocycles.

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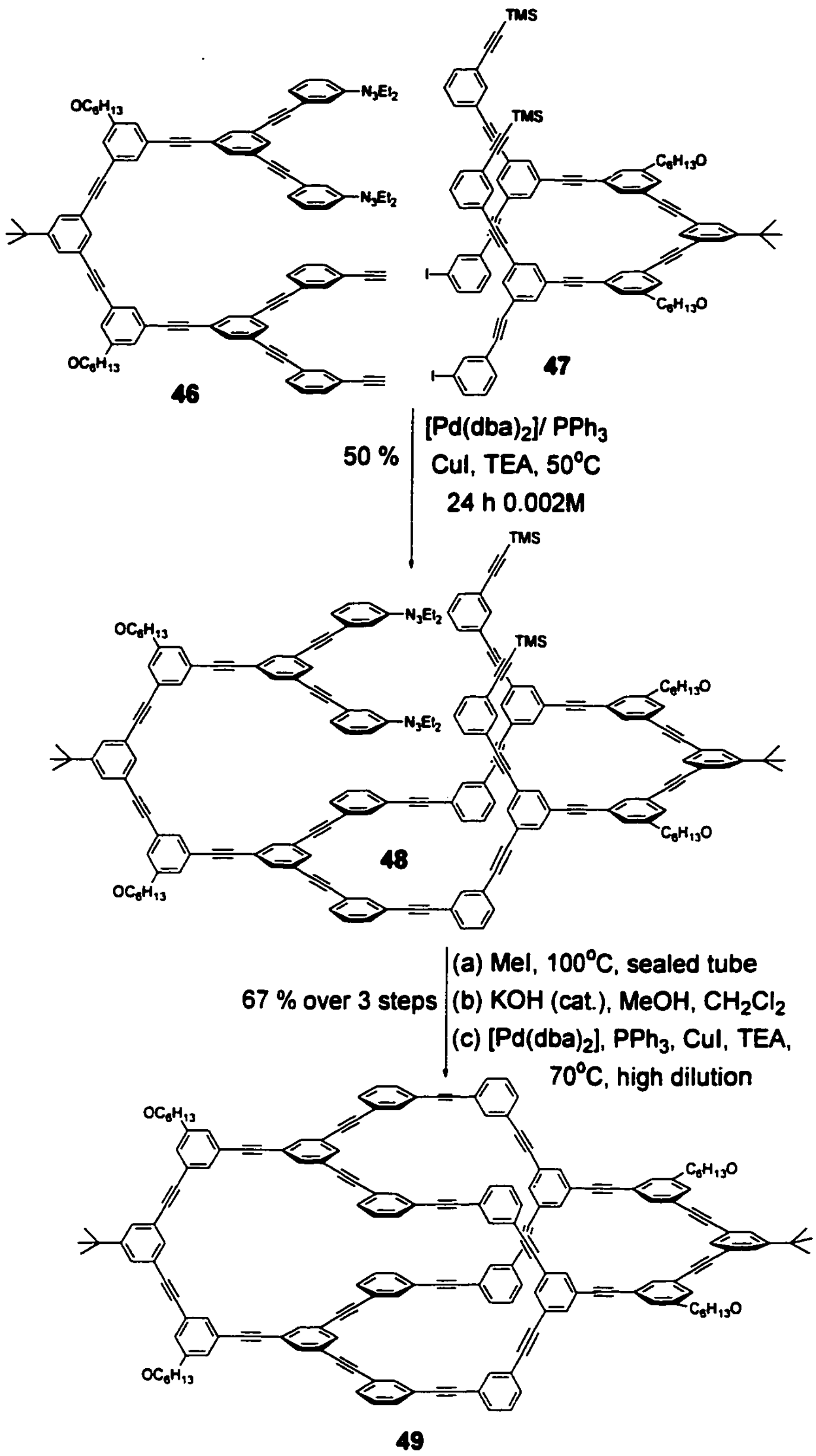
[31] Moore, J.S. *Acc. Chem. Res.* **1997**, *30*, 402.

[32] (a) Wu, Z.; Lee, S.; Moore, J. S. *J. Am. Chem. Soc.* **1992**, *114*, 8730-8732. (b) Yu, Z.; Kahr, M.; Walker, K. L.; Wilkins, C. L.; Moore, J. S. *J. Am. Chem. Soc.* **1994**, *116*, 4537-4550.



Scheme 14. Nanostructure construction via double intramolecular cyclization strategies.

Moore also used this approach to synthesize the macrotetracycle, **49.**[33] Although a number of isomeric nanostructures of **49** could be prepared, the order at which various sites are deprotected and coupled allows for exquisite control over which macrocycle is formed (Scheme 14).

Perhaps the greatest synthetic challenge posed by a 3-dimensional macrocycle is the controlled laboratory synthesis of fullerenes. Since the discovery of a third allotrope of carbon by Smalley and Kroto,[34] fullerene chemistry has become a prolific topic of research. The unexpected physical properties of C_{60} include superconductivity of its alkali metal salts (ie. K_3C_{60} (T_c = 18 K)), soft ferromagnetism (T_c = 16 K) and a large third-order optical nonlinearity ($Z^{(3)} = 7 * 10^{-12} \bullet 6 * 10^{-8}$ esu). [35] Some highly functionalized C_{60} adducts have been observed to exhibit electroluminescence and have been dubbed "Bucky Light Bulbs".[36] These results are especially surprising due to the fact that C_{60} itself is a known luminescence quencher. It has been postulated that an even greater variety of physical and chemical properties exist for endohedral metallofullerenes, specifically C_{60}, due to its high symmetry. These endohedral host-guest complexes represent one of the most complicated and advanced spherical hosts know to date.[37] Fullerenes are currently prepared by the graphite evaporation method, but suffers from a lack of size specificity and lengthy separation and purification. The endohedral complexes of C_{60} have yet to be prepared in quantities in excess of a milligram and only lanthanides, Ca, Sr, Ba have been included within the empty shell.[38] However, a scandium carbide endohedral metallofullerene $(Sc_2C_2)@C_{84}$ has been prepared (3.5 mg) and isolated as a black powder.[39]

The development of a general synthetic approach to fullerenes should address the limitations of the current graphite evaporation method and allow for greater versatility in

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[33] Moore J.S. In *Modern Acetylene Chemistry;* Diederich, F.; Stang, P.J., Eds.; Wiley-VCH, Weinheim, Germany, 1995; pp 415-441.

[34] Kroto, H.W.; Heath, J.R.; O'Brien, R.F.; Curl, R.E.; Smalley, E. *Nature*, **1985**, *318*, 162.

[35] *The Fullerenes*; Kroto, H.W.; Fischer, J.E.; Cox, D.E., Eds.: Pergamon Press, New York, **1993**.

[36] Hutchison, K.; Gao, J.; Schick, G.; Rubin, Y.; Wudl, F. *J. Am. Chem. Soc.* **1999**, *121*, 5611.

[37] (a) MacGillivray, L.R.; Atwood, J.L. *Angew. Chem. Int. Ed. Engl.* **1999**, *38*, 1018. (b) Seel, C.; Vogtle, F. *Angew. Chem. Int. Ed. Engl.* **1992**, *31*, 528.

[38] Kubozono, Y.; Maeda, H.; Takabayashi, Y.; Hiraoka, K.; Nakai, T.; Kashino, S.; Emura, S.; Ukita, S.; Sogabe, T. *J. Am. Chem. Soc.* **1996**, *118*, 6998.

[39] Wang, C-R.; Kai, T.; Tomiyama, T.; Yoshida, T.; Kobayashi, Y.; Nishibori, E.; Takata, M.; Sakata, M; Shinohara, H. *Angew. Chem. Int. Ed. Engl.* **2001**, *40*, 397.

metal incorporation. Furthermore, a direct synthetic approach would shed light on the crucial stages of fullerene formation and the rules that may govern the observed product distributions. This process may ultimately allow for the design of more sophisticated fullerene precursors and derivatives thereof.

Several strategies have been proposed for the synthesis of fullerenes based on the "zipping-up" of carbon shells. These approaches may allow inclusion of metals within the shell. Endohedral fullerenes may also be prepared through "cracking" open of the shell and insertion of the metal ion, and reformation of the carbon host.[40] While investigating the selective six-fold functionalization of C_{60},[41] Rubin and co-workers found that heating of C_{60} at 55 °C for 4 days in the presence of a rigid diazobutadiene system created an opening in the surface of the carbon sphere.[42] Consequently, small amounts of helium were found to be capable of entering the orifice and becoming lodged within the cavity.[43] So far, the group has not yet re-stitched the carbon network, to trap the atom within the fullerene. Similarly, Rubin's group have applied their opening protocol to the synthesis of a non-classical fullerene C_{62} which incorporates an anti-aromatic four membered ring within the structure.[44]

An attractive synthetic route to carbon rich materials and fullerenes lies with the use of strained cycloalkynes. As starting materials, these structures possess large amounts of energy, which upon certain stimuli, could lead to coalescence of the cyclic structure to a thermodynamically more stable carbon sphere. The use of an "explosion" of a strained precursor has the potential to produce carbon rich materials[45] such as bucky-tubes (closed shelled carbon nanotubes), bucky onions, and bucky balls (fullerenes). Incorporation of cyclopentadienyl or benzenoid moieties would form interesting acetylenic cyclophanes with the possibility to co-ordinate metal fragments.[46]

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[40] Rubin, Y. *Chem. Eur. J.* **1997**, *3*, 1009.
[41] Qian, W.; Rubin, Y. *Angew. Chem. Int. Ed. Engl.* **1999**, *38*, 2356.
[42] Schick, G.; Jarrosson, T.; Rubin, Y. *Angew. Chem. Int. Ed. Engl.* **1999**, *38*, 2360.
[43] Rubin, Y.; Jarrosson, T.; Wang, G-W.; Bartberger, M.D.; Houk, K.N.; Schick, G.; Saunders, M.; Cross, R.J. *Angew. Chem. Int. Ed. Engl.* **2001**, *40*, 1543.
[44] Qian, W.; Barberger, M.D.; Pastor, S.J.; Houk, K.N.; Wilkins, C.L.; Rubin, Y. *J. Am. Chem. Soc.* **2000**, *122*, 8333.
[45] Birkett, P.R.; Terrones, M. *Chem. Brit.* **1999**, 45.
[46] Faust, R. *Angew. Chem. Int. Ed.* **1998**, *37*, 2825.

Diederich first observed that that under Fourier transform laser desorption (FT-LD) mass spectrometric experiments that the cations of cycloalkynes **50** and **51** rearranged to give spherical C_{60}^+ cations. The preparation of bulk quantities of the cycloalkyne precursors have remained elusive.[47] The use of synthetically accessible "cyclophynes" or phenyl-acetylene cyclophanes can also rearrange to bucky-products.

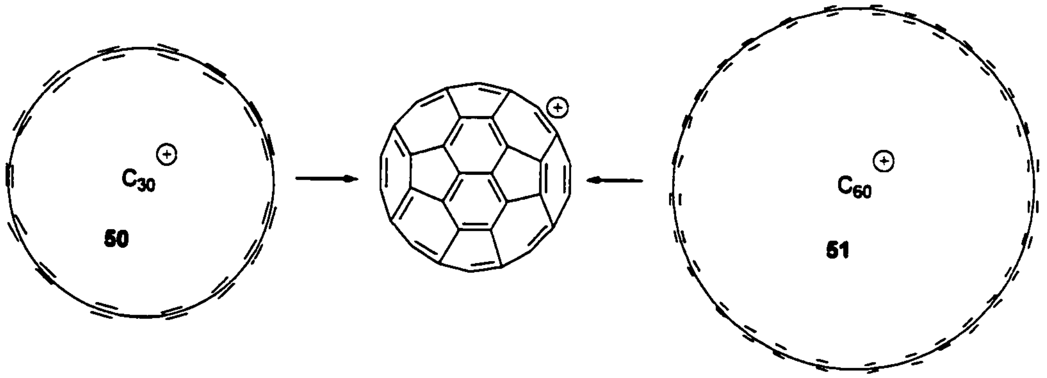
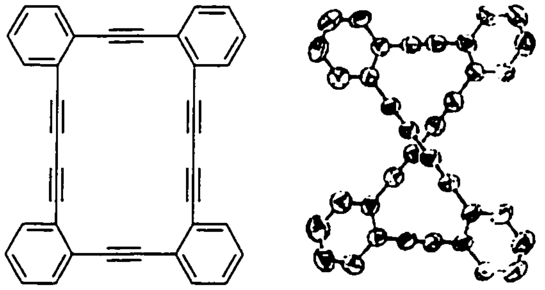


Figure 8. Transformation of cationic cycloalkyne carbon rings to form fullerenes.

These cyclophanes have emerged as the most prevalent precursors to carbon rich materials. Vollhardt and co-workers have synthesized the cycloalkyne, **52**, whose X-ray analysis revealed it adopted a non-planar, twisted D_2-symmetric conformation in the crystal in which the two butadiynyl bridges cross on top of one another.[48]



52

Figure 9. The structure of 52 actually exhibits a twisted helical conformation in the crystal.

When **52** was heated to 245 °C, it "explodes violently with a flash of orange light" producing a carbon residue. Analysis by transmission electron microscopy (TEM) revealed the presence of amorphous carbon and graphite and closed shell carbon

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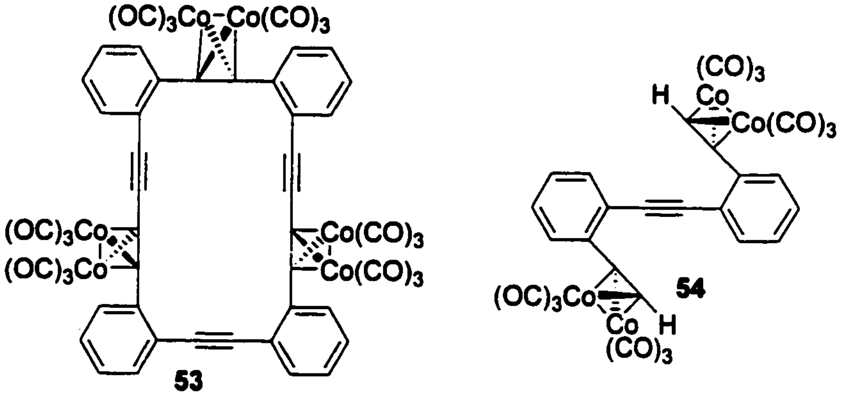
[47] (a) Rubin, Y.; Kahr, M.; Knobler, C.B.; Diederich, F.; Wilkins, C.L. *J. Am. Chem. Soc.* **1991**, *113*, 495. (b) McElvany, S.W.; Ross, M.M.; Goroff, N.S.; Diederich, F. *Science*, **1993**, *259*, 1594.

[48] Boese, R.; Matzger, A.J.; Vollhardt, K.P.C. *J. Am. Chem. Soc.* **1997**, *119*, 2052.

particles, bucky-onions. Vollhardt and co-workers have since reported an extension of their studies. They describe the reaction of cobalt carbonyl complexes **53** and **54** among others to give carbon nanotubes.[49] Upon heating these compounds, a smooth reaction commences where the compounds release CO gas. Heating above 200 °C, caused an irreversible polymerization and spectral and GC analysis revealed the loss of CH_4, H_2, and CO_2 gases. TEM analysis of the pyrolytic powders showed that while graphitization occurs at low temperatures, increased heating (800 °C, 6 h) produced large quantities of well-formed carbon onions and multiwalled nanotubes with very little amorphous carbon. Metal was deposited in crystalline form inside the tubes and onions and occasionally at the tips. These studies suggest that a specific molecular packing is necessary for closed shell carbon construction.



Scheme 15. Cobalt carbonyl complexes of carbon networks that form metal encapsulated nanostructures.

Consequently, others have begun to investigate carbon rich organometallic materials. Bunz *et al.* were interested in tetragonal cyclobutadiene complexes featuring the tetraethynylcyclobutadiene motif, **55** (Scheme 16), due to its postulated importance in fullerene formation.[50] Using a palladium cross-coupling approach, Bunz and co-workers assembled cyclophane **57** in good yield but additional alkyl substituents were needed to grow quality single crystals. X-ray crystal analysis revealed that the hydrocarbon deviated from planarity. The two planes defined by the four alkynyl groups, are tilted 13° from the central cyclobutadiene ring (Figure 10). The resulting concave shape was also established by semiemperical computer calculations and is not due to crystal packing

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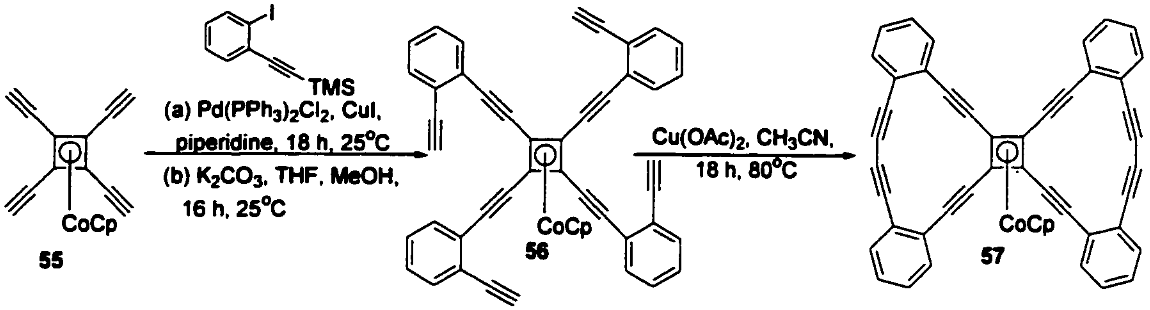
[49] Dosa, P.I.; Erben, C.; Iyer, V.S.; Vollhardt, K.P.C.; Wasser, I.M. *J. Am. Chem. Soc.* **1999**, *121*, 10430.
[50] Laskoski, M.W.; Roidl, G, Smith, M.D.; Bunz, U.F. *Angew. Chem. Int. Ed. Engl.* **2001**, *40*, 1460.

effects or interactions. In addition, the central cyclobutadiene ring is rectangular and not square, which supports the notion that two aromatic dehydroannulenes are fixed onto the ring.



Scheme 16. Organometallic hydrocarbons as potential endohedral carbon rich material precursors.

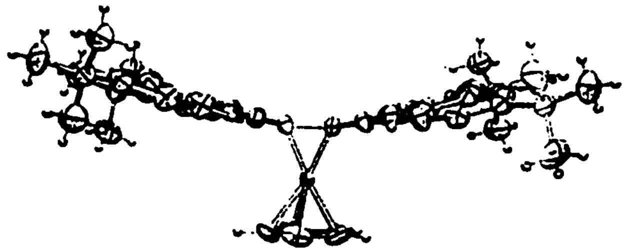


Figure 10. X-ray crystallographic analysis of a cobalt carbonyl cyclobutadienyl cyclophyne shows a concave butterfly like geometry.

Haley and Langsdorf have tried to trap strained cycloalkynes or cyclophynes as an octacobalt complex in order to assemble the skeleton quickly and efficiently using known methodology.[51] Subsequent efficient and mild removal of the transition metal complexes should give the product, **61**. Unfortunately, upon iodine promoted decomplexation, the dppm ligand was the sole organic soluble component isolated, although a large amount of insoluble material was also recovered.

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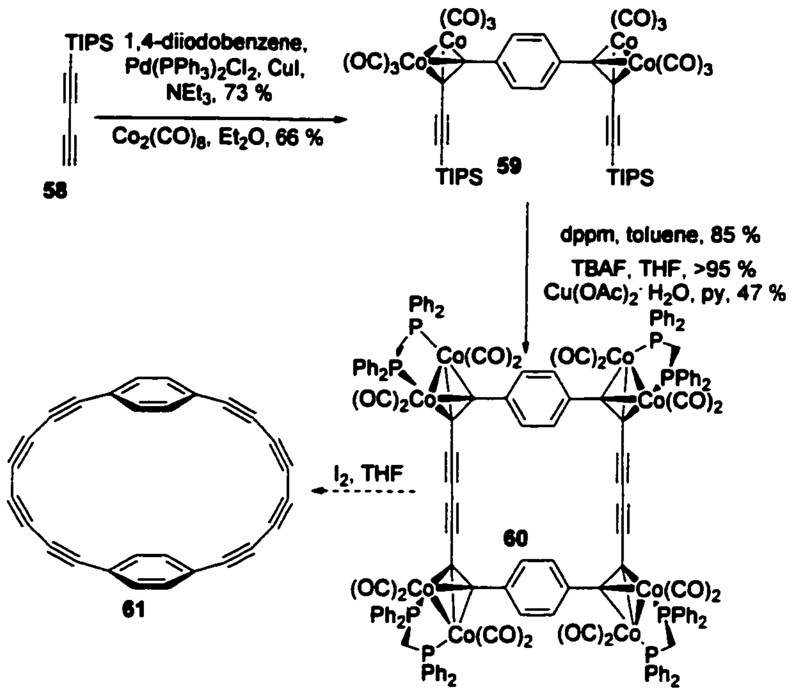
[51] Haley, M.M.; Langsdorf, B.L. *Chem. Commun.* **1997**, 1121.



*Scheme 17. Proposed synthesis of [8.8]paracyclophaneoctayne, **61**.*

Rubin and Tobe have each independently made major steps towards the transformation of acetylenic cyclophanes into fullerenes through the synthesis of sixty-carbon cyclophyne cages. Under appropriate activation, the cages should "explode" and colapse to buckminsterfullerene, C_{60}. Rubin's prototypical cyclophyne **64**, was synthesized in a straightforward manner described in Scheme 18.[52] X-ray quality crystals of macrocycle, **64**, were grown in ODCB (*o*-dichlorobenzene) and displayed a D_3-symmetric helical conformation. Interestingly, within the crystal packing structure it is revealed that each pair of like-handed helices stack along the *z*-axis but are separated from each other by a channel of ODCB molecules. Racemization of the enantiomeric forms should proceed *via* a dynamic process in which the two arene planes must rotate in opposite directions. The energy of the transition state for this rotation was estimated at 3-6 kcal/mol, suggesting that even at low temperature the racemization must occur very rapidly. Unfortunately, MALDI-TOF FTMS negative ion mass spectroscopy revealed that **64** did not rearrange to C_{60}, although an intermediate $C_{60}H_{14}$ was observed and implied that either **64** was relunctant to lose the remaining hydrogen atoms or the molecule possessed too much flexibility to undergo the required controlled reaction

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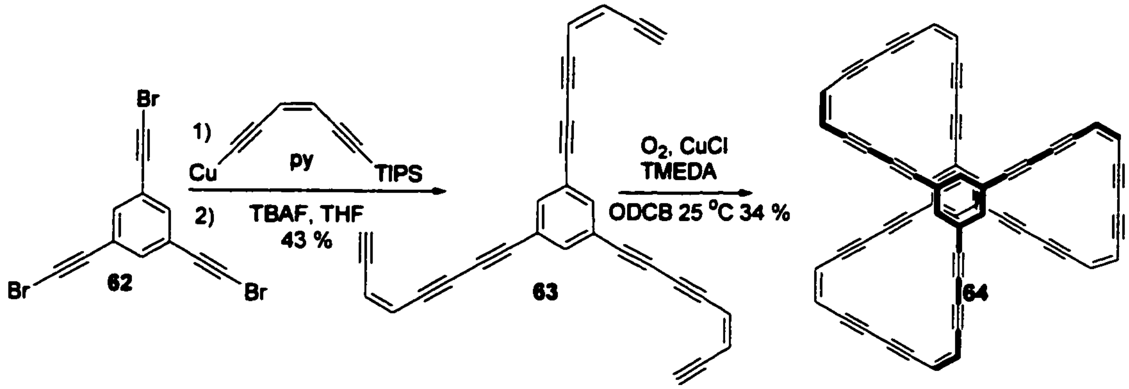
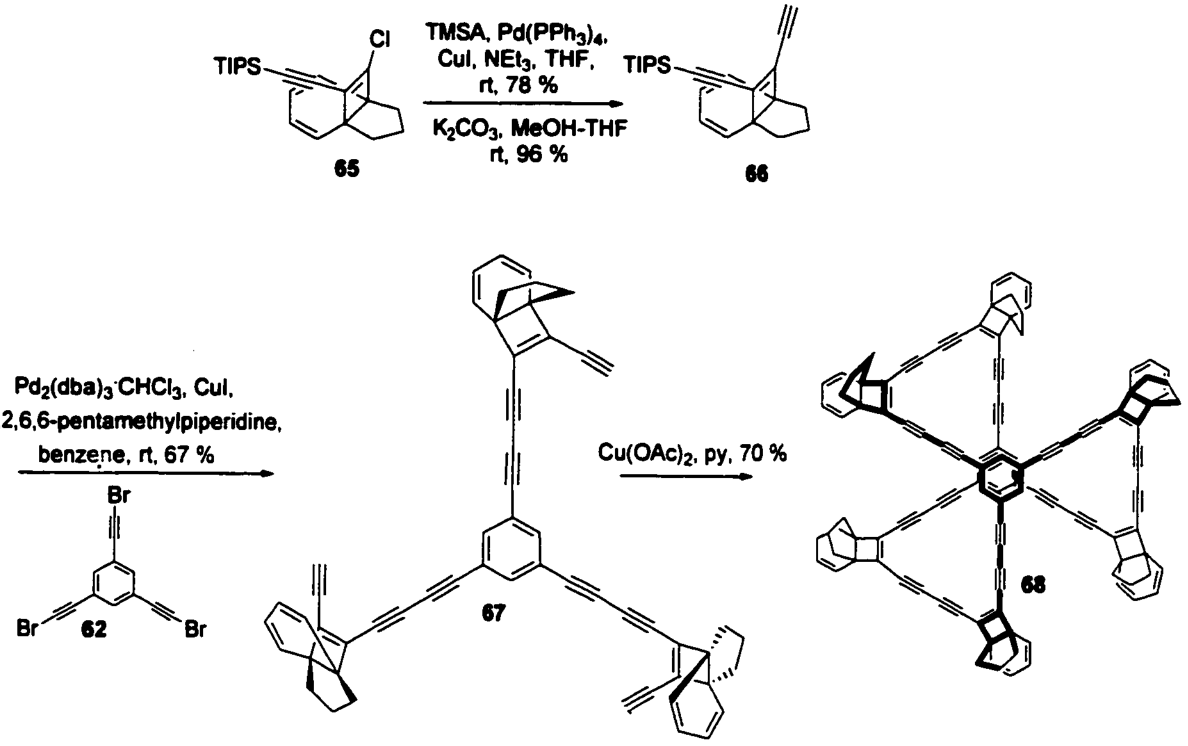
[52] Rubin, Y.; Parker, T.C.; Khan, S.I.; Holliman, C.L.; McElvany, S.W. *J. Am. Chem. Soc.* **1996**, *118*, 5308.

pathways. In addition, attempts to induce rearrangement through pyrolysis or in solution by treatment with laser irradiation did not produce any fullerene.



*Scheme 18. Rubin's synthesis of a flexible acetylenic cyclophane, $C_{60}H_{18}$, **64**.*



*Scheme 19. Tobe's synthesis of a rigid acetylenic cyclophane, $C_{60}H_6(Ind)_6$, **68**.*

Tobe and co-workers have proposed the use of a [2+2] cycloreversion of [4.3.2]propellatriene derivatives to generate reactive polyynes.[53] The preparation of [16.16.16](1,3,5)cyclophanetetracosayne as a precursor to C_{60} fullerene is described in

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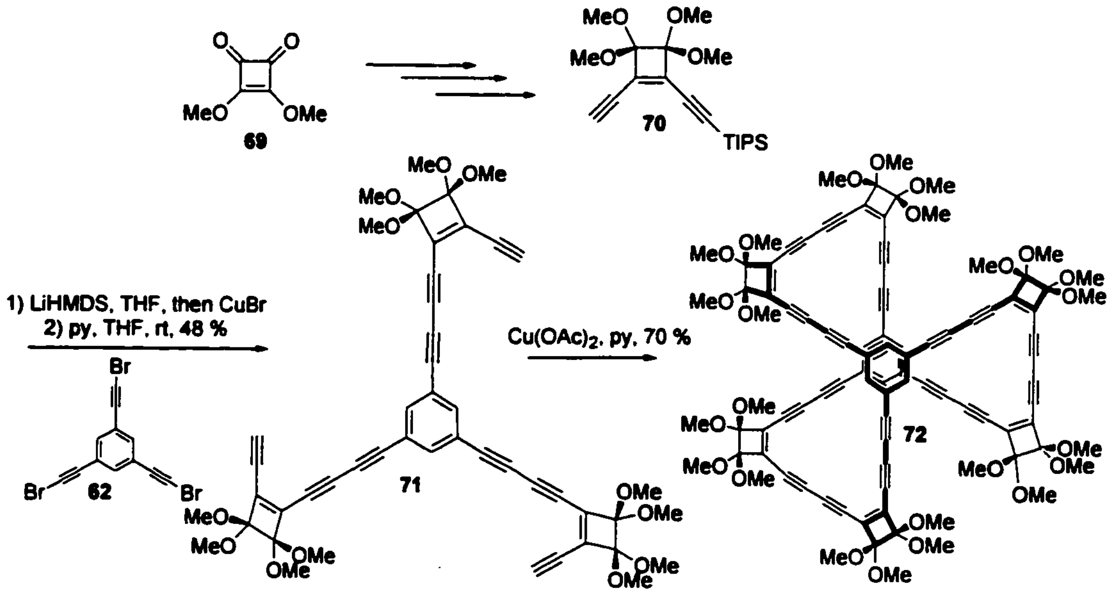
[53] Tobe, Y.; Fuji, T.; Matsumoto, H.; Tsumuraya, K.; Noguchi, D.; Nakagawa, N.; Sonoda, M.; Naemura, K.; Achiba, Y.; Wakabayashi, T. *J. Am. Chem. Soc.* **2000**, *122*, 1762.

Scheme 19.[54] Extrusion of six aromatic indane fragments from **68** would generate $C_{60}H_6$, a proposed precursor to fullerenes. The laser desorption time-of-flight mass spectrum of **68** in both the positive and negative ion modes showed coalescence to C_{60} and no larger clusters of peaks corresponding to larger fullerenes were observed. It is important to note that although this does not represent an actual laboratory synthesis, it does represent the first size selective formation of fullerenes.



Scheme 20. Rubin's use of acetylenic cyclophanes as fullerene precursors.

Rubin's macrocycle, **64** did not lose hydrogen in the gas phase and has modified the enediyne portion of the molecule. Rubin's synthesis of the cyclobutenedione cyclophane, **72**, is depicted in Scheme 20.[55] The cyclobutenedione moiety was found to release sensitive acetylene bonds in the gas phase with concomitant loss of carbon monoxide.[56] Rubin and co-workers then observed that the acetylenic cyclophane underwent coalescence to C_{60} ions in the Fourier transform ion cyclotron resonance laser desorption mass spectra in both the negative and positive ion modes, although the ion fragments appear more intense in the former spectrum.

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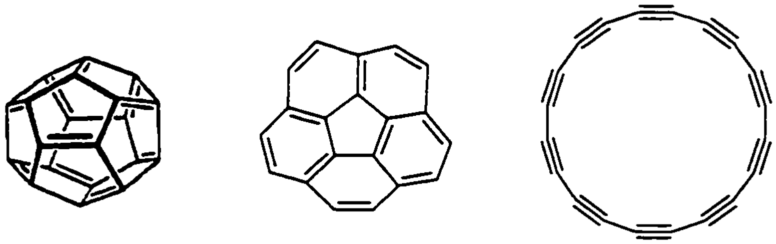
[54] Tobe, Y.; Nakagawa, N.; Naemura, K.; Wakabayashi, T.; Shida, T.; Achiba, Y. *J. Am. Chem. Soc.* **1998**, *120*, 4544.

[55] Rubin, Y.; Parker, T.C.; Pastor, S.J.; Jalisatgi, S.; Boulle, C.; Wilkins, C.L. *Angew. Chem. Int. Ed. Engl.* **1998**, *37*, 1226.

[56] Rubin, Y.; Lin, S.S.; Knobler, C.B.; Anthony, J.; Boldi, A.M.; Diederich, F. *J. Am. Chem. Soc.* **1991**, *113*, 6943.

The obvious promise of cycloalkynes in fullerene synthesis has not hindered the minds of organic chemists to imagine new routes for their construction. In early 2000, Prinzbach and co-workers synthesized the smallest fullerene ever detected.[57] The elusive C_{20} structure is a non-natural fullerene not observed from the products of the graphite vaporization method. The synthesis was approached from dodecahedron ($C_{20}H_{10}$) a saturated hydrocarbon cage have 12 pentagon faces (Figure 11). An exhaustive bromination procedure was used to strip off the hydrogens and replace them with bromine atoms. Electron-impact mass spectroscopy detected the debrominated products as well as the production of C_{20}^{+} and C_{20}^{2+}. To exclude the possibility that the cage had rearranged in the gas phase, a bowl-like isomer, corannulene, was subjected to the same bromination procedure the electron-impact mass spectra was compared to that of C_{20} and a cycloalkyne "ring" precursor. These comparisons provide strong evidence for the first synthesis of C_{20}.


C_{20} Cage

$C_{20}H_{10}$ Bowl

C_{20} Ring

Figure 11. The various forms of C_{20}.

Ferrier has proposed two novel routes to C_{60} dubbed the "tennis ball" and "basket-and-lid" routes.[58] The strategy involves the synthesis of large 60-carbon frameworks that consist of a trindane repeating unit. It is proposed that upon dehydrogenation these compounds would fold in a manner to form C_{60} analogous to folding the material and forming the sphere of tennis ball (Figure 11). In addition, it is also imaginable to form as an intermediate bucky-bowl, where the folding is stopped at an advanced stage and a basket-shaped hydrocarbon would remain with the last flap unattached and acting as a lid.

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[57] Prinzbach, H.; Weller, A.; Landenberger, P.; Wahl, F.; Worth, J.; Scott, L.T.; Gelmont, M.; Olevano, D.; v. Issendorff, B. *Nature (London)* **2000**, *407*, 6800.

[58] Ferrier, R.J.; Holden, S.G.; Gladkihk, O. *J. Chem. Soc., Perkin Trans. I* **2000**, 3505.

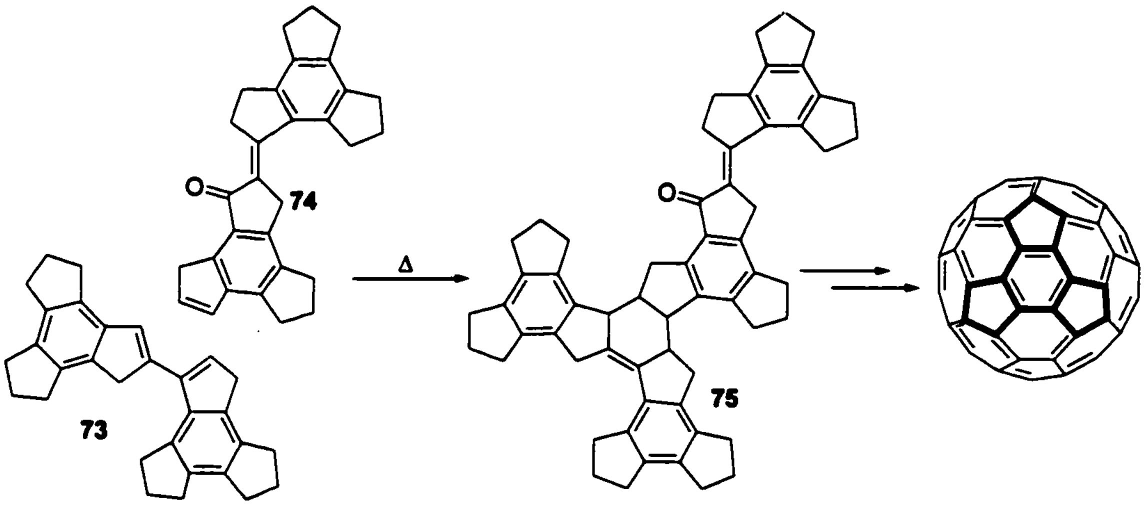
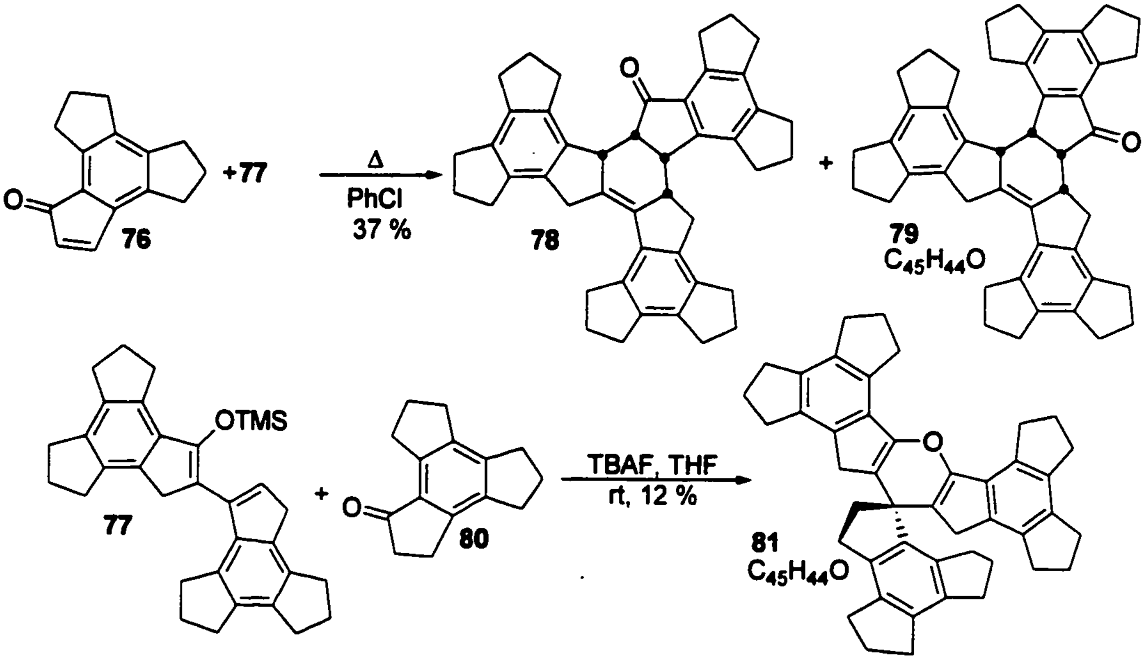


Figure 12. Large carbon networks featuring the trindane unit may serve as precursors to fullerenes.

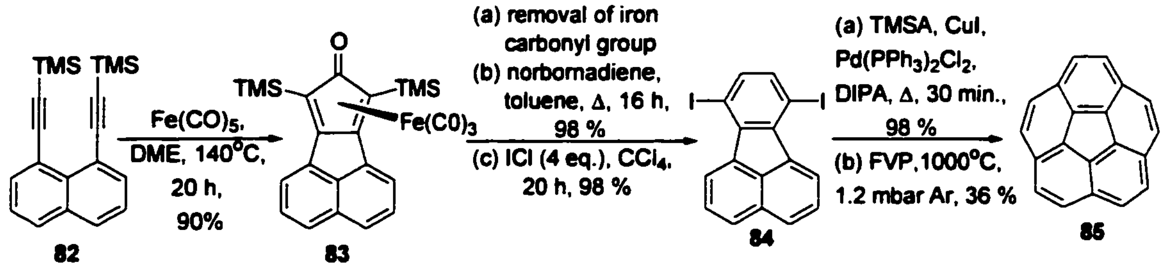
Although development of the route is in its infancy, Ferrier and co-workers have managed to construct two relevant C_{45} compounds and have demonstrated the flexibility and usefulness of the trindane unit (Figure 12).



Scheme 21. Synthesis of relevant C_{45} units containing repeating trindane units.

Other research groups have centered their research on the synthesis of bucky-bits or segments of the fullerene skeleton. Accordingly, the simplest of these bits, corannulene ($C_{20}H_{10}$), has received increased interest resulting in a novel synthesis. Knolker and co-workers reported a short construction of **85** using an iron-mediated

[2+2+1] cycloaddition (Scheme 22).[59] As with many of the existing syntheses, the final step involves high temperatures, typically flash vacuum pyrolysis (FVP) to initiate cyclization.



Scheme 22. Synthesis of the "bucky-bit" corannulene via *an iron transition metal complex.*

Mehta *et al.* have also studied routes to expanded fragments of the fullerene sphere.[60] The larger fragments begin to bend, (ie. show distinct curvature and are termed bucky-bowls). Each of the surfaces of these structures is expected to exhibit markedly different chemical reactivity which is in contrast to their planar analogues. Again, FVP is the key step in forming the five-membered rings and transforming aromatic precursors into bucky-bowls. Unfortunately, FVP of **89** only succeeded in forming one of the three necessary five-membered rings giving **90** in 20% yield (Scheme 23)

Recently however, novel milder conditions have evolved for the construction of semibuckminsterfullerenes.[61] Rabideau *et al.* have had success in obtaining **93** as a fragment of the C_{60} skeleton.[62] Their procedure takes advantage of the propensity for carbenoids to undergo coupling to form aromatic rings (Scheme 24). Bucky-bowl, **93**, also allowed the first X-ray crystallographic analysis of a semibuck-minsterfullerene. The molecules stack in a concave to convex fashion. The concave surface also cocrystallizes with two molecules of dicholoromethane and as a result, contact between the hydrocarbon moieties is minimized.

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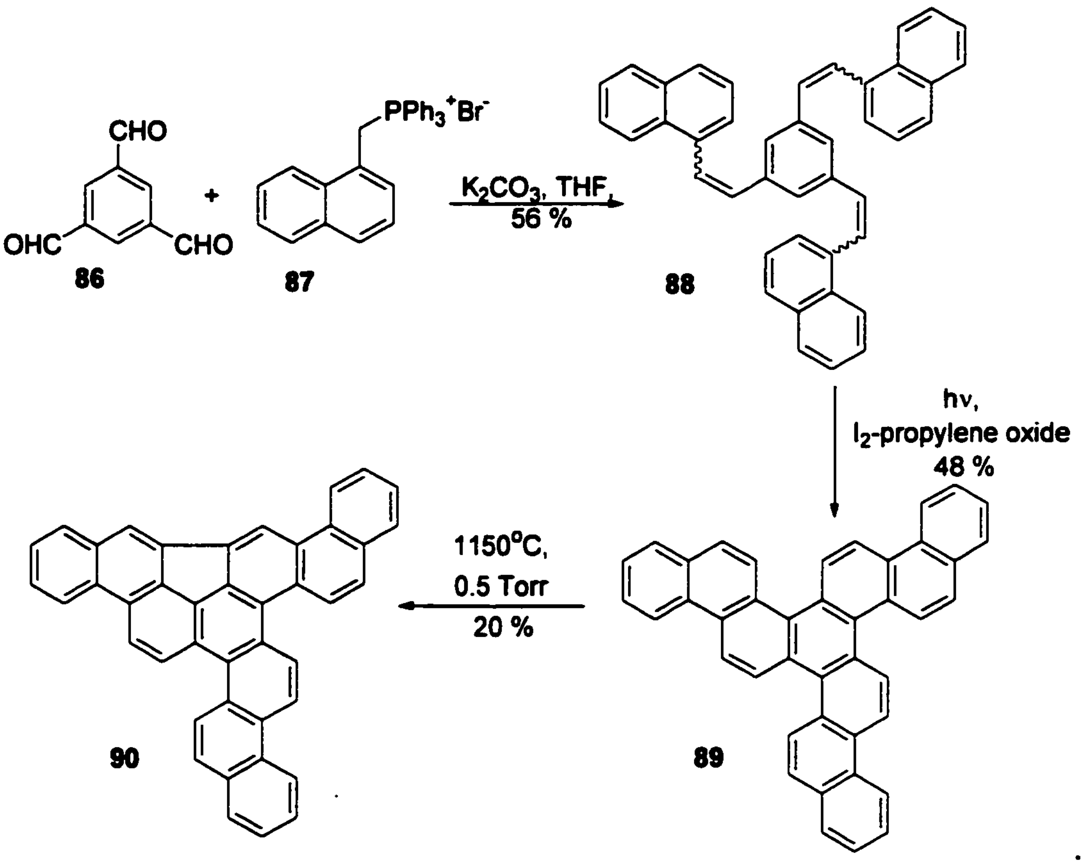
[59] Knolker, H.-J.; Braier, A.; Brocher, D.J.; Jones, P.G.; Piotrowski, H. *Tetrahedron Lett.* **1999**, *40*, 8075.

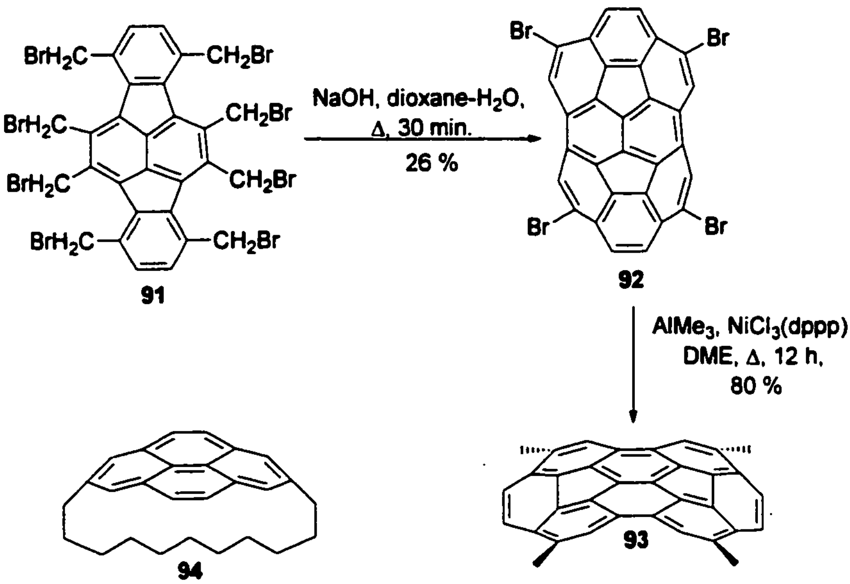
[60] Mehta, G.; Panda, G.; Shah, S.R.; Kunwar, A.C. *J. Chem. Soc., Perkin Trans. 1* **1997**, 2269.

[61] (a) Sygual, A.; Rabideau, P.W. *J. Am. Chem. Soc.* **1999**, *121*, 7800. (b) Sygula, A.; Xu, G.; Marcinow, Z.; Rabideau, P.W. *Tetrahedron* **2001**, *57*, 3637.

[62] Sygula, A.; Marcinow, Z.; Fronczek, F.R.; Guzei, I.; Rabideau, P.W. *Chem. Commun.* **2000**, *24*, 2439.



Scheme 23. Towards a synthesis of C_3*-tribenzohemifullerene, a* $C_{42}H_{18}$ *fragment of* C_{60}.

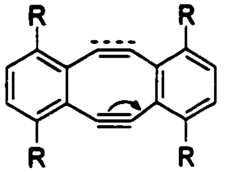


Scheme 24. A novel synthetic route to a crystalline bowl shaped semibuckminsterfullerene, ***93*** *and the angular pyrenophane,* ***94****.*

Bodwell and co-workers have synthesized novel non-planar aromatic compounds as models to study the surface reactivity of fullerenes. Their protocol allows one to build paracyclopyrenophanes that are tethered at either end by a linker that forces the normally planar hydrocarbon to bend and alter into shape.[63] X-ray crystallographic analysis has confirmed the angular conformation of **94** and Diels-Alder studies have begun to reveal the interesting reactivity of these surfaces (Scheme 24).[64]

1.4 Carbon Networks and Cyclophanes Bearing Strained Acetylenes

The hybridization of the sp carbon-carbon bond dictates a linear geometry, however, this bond can often be bent or stretched more than sp^3 or sp^2 hybridization centers. The normal linear geometry for the C-C≡C bond is often severely distorted (165-155°) from the planar value. Krebs has openly proposed that deviations from linearity that are not greater than 10° occur with little changes in energy.[65] Sondheimer's early work with strained acetylenes concerned the syntheses of cycloalkynes in the form of dehydrobenzoannulenes. His studies and syntheses of the dehydrodibenzocyclooctenes and –ynes showed that these surprisingly stable substances were connected by triple bonds whose angles ranged from 165° to below 154°! (Figure 13).[66,67,68]



95 R = H, alkyne, 155.8°
96 R = H, alkene, 154.0°
97 R = Me, alkene, 165.8°

Figure 13. Sondheimer's early investigations into strained acetylene linkages.

Sondheimer's work perhaps laid the groundwork for the current resurgence in strained acetylene chemistry. Strained polyynes are now viewed as the prime targets for the synthesis of carbon nanostructures *via* the topochemical polymerization or rearrangement of alkynes. Recently, strained diynes systems have also emerged in the

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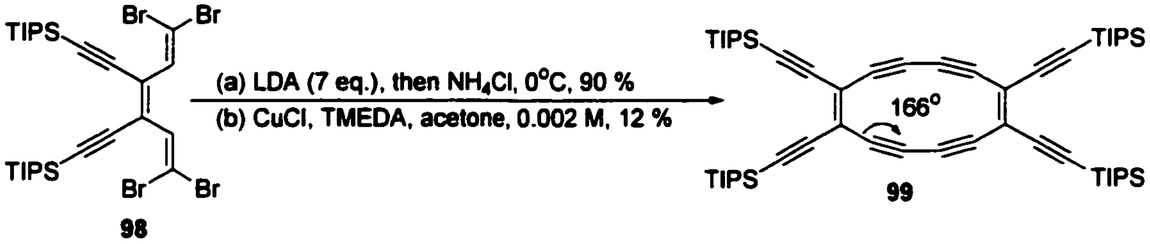
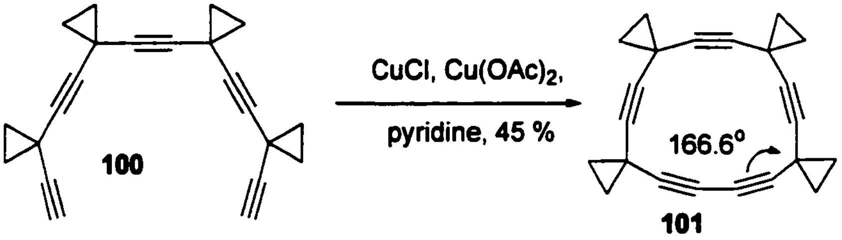
[63] Bodwell, G.J.; Miller, D.O.; Vermeij, R.J. *Org. Lett.* **2001**, *3*, 2093.
[64] Bodwell, G.J.; Fleming, J.J.; Mannion, M.R.; Miller, D.O. *J. Org. Chem.* **2000**, *65*, 5360.
[65] Krebs, A; Wilke, J. *Top. Curr. Chem.* **1983**, *109*, 189.
[66] Destro, R.; Pilati, T.; Simonetta, M. *J. Am. Chem. Soc.* **1975**, *97*, 658.
[67] De Graaff, R.A.G.; Gorter, S.; Romers, C.; Wong, H. N.; Sondheimer, F. *J. Chem. Chem. Soc. Perkin II* **1981**, 478.
[68] Au, M.-K.; Siu, T.-W.; Mak, T. C.; Chan, T.-C. *Tetrahedron Lett.* **1978**, 4269.

study of novel carbon allotropes. In a communication aptly dedicated in memorium to Sondheimer, Diederich and co-workers reported the stable synthesis of [12]- and [18]-annulenes from tetratethynylethene and reported deviated mean angles of ~166° from planarity for the acetylenes (Scheme 25).[69]

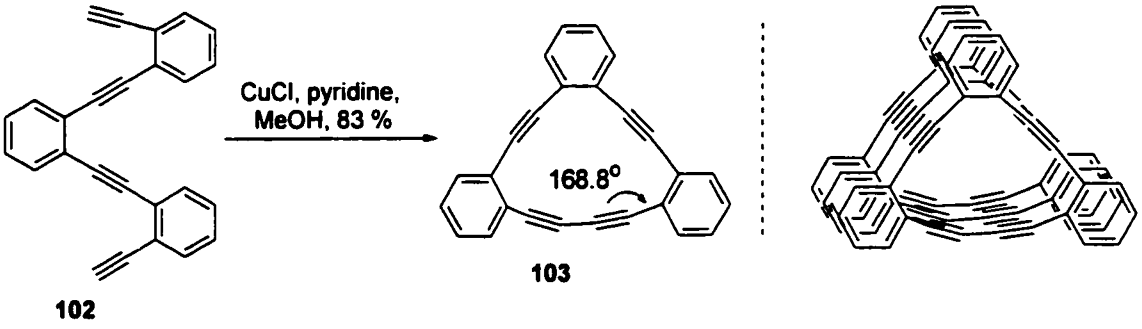


Scheme 25. Novel tetraethynylethene dimer synthesized by Diederich.

The perspirocyclopropanated pentaynes synthesized by de Meijere, Scott, Smith and co-workers also exhibited strained acetylene bonds with angles of 166° (Scheme 26).[70] More importantly, macrocycle, **101**, exhibited through space π-orbital interactions which caused bathochromic shifts in the long wavelength UV spectra, relative to certain linear and cyclic standards.



Scheme 26. Novel perspirocyclopropanated pentayne synthesized by de Meijere.



*Scheme 27. The synthesis of a strained dehydrobenzoannulenes, **103** (left) and a figure depicting its packing in the solid state.*

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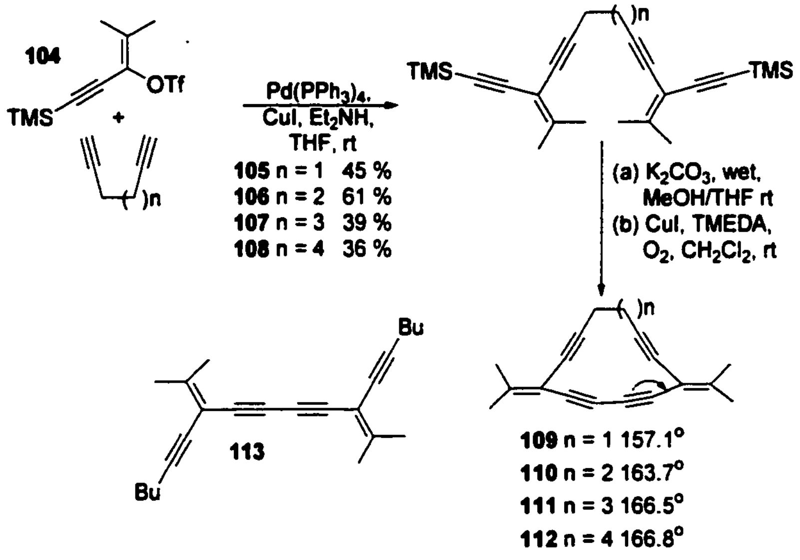
[69] Anthony, J.; Knobler, C. B.; Diederich, F. *Angew. Chem. Int. Ed.* **1993**, *32*, 406.

[70] Scott, L. T.; Cooney, M. J.; Otte, C.; Puls, C.; Haumann, T.; Boese, R.; Carroll, P. J.; Smith, A. B.; de Meijere, A. *J. Am. Chem. Soc.* **1994**, *116*, 10275.

Vollhardt and co-workers revisited strained DBAs and synthesized **103** as a dehydro[14]annulene.[71] The X-ray crystal packing structure revealed a motif amenable to topochemical polymerization. The molecules of **103** were stacked on top of each other but slightly offset (Scheme 27). Vollhardt and co-workers also proposed the structure of a trimeric unit from a proposed topochemical polymerization.

Tykwinski and co-workers synthesized a series of dendralenes to relate structural and spectroscopic attributes as a function of ring strain.[72] The X-ray crystal structures clearly depict the deformed angle of the butadiyne linkages in the dendralenes. The observed shifts in the Raman and ^{13}C NMR spectra of compounds **109-112** showed trends that directly reflect the varying electronic structure of the molecules. The trends in the spectroscopic investigation were compared against a linear standard, **113** (Scheme 28). The studies showed that despite the ring strain apparent in **109** and **110**, it played a small role in varying the electronic absorption spectra



Scheme 28. The synthesis of a series of strained dendralenes.

Tsuji *et al.* were able to induce strain onto alkynes and diynes in the process of constructing novel *meta*-phenyl acetylene cyclophanes (Scheme 29).[73] In order to build the macrocycles using a dimerization strategy, the terminal acetylene groups of **113** were

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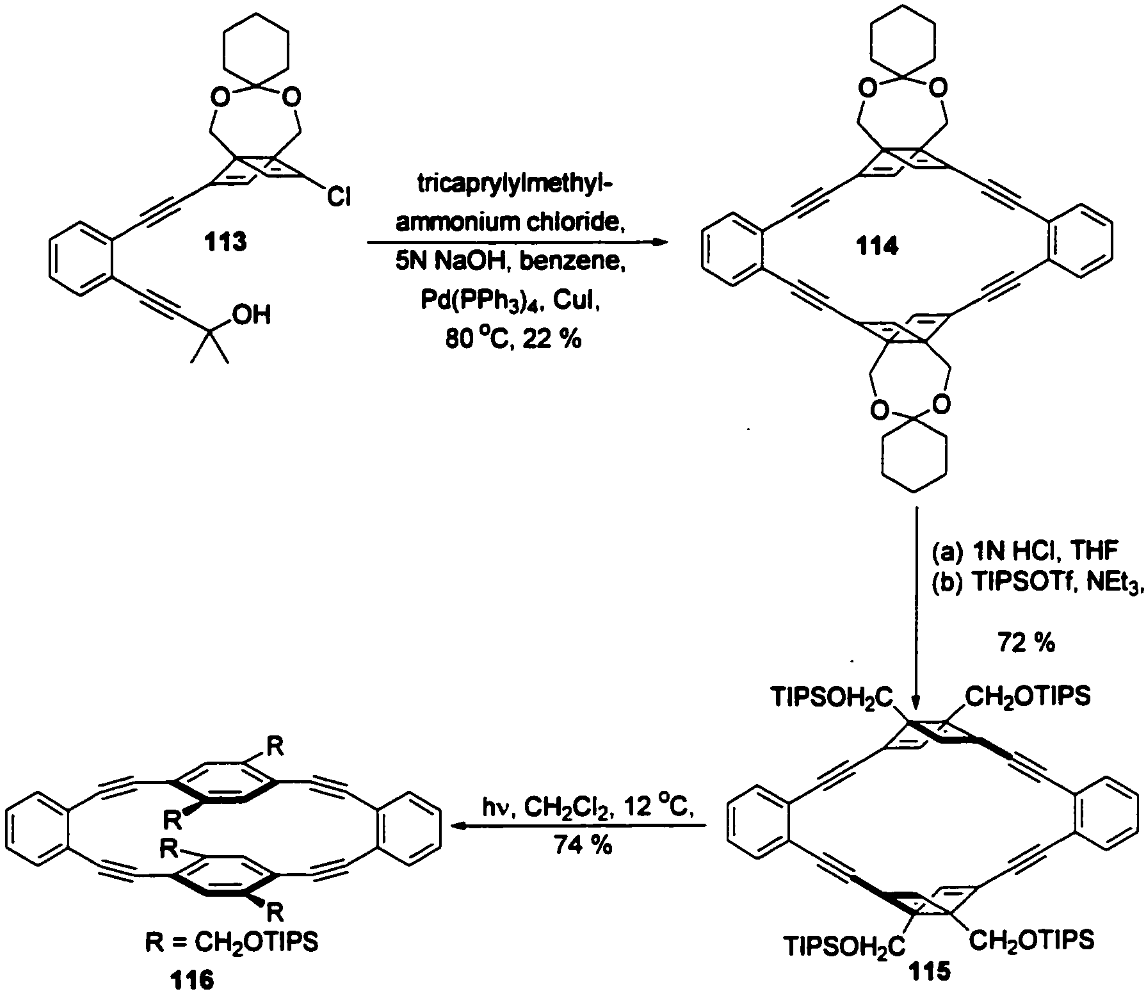
[71] Baldwin, K. P.; Matzger, A. J.; Scheiman, D. A.; Tessier, C. A.; Volhardt, K. P. C; Youngs, W. J *Synlett* **1995**, 288.

[72] Eisler, S.; McDonald, R.; Loppnow, G. R.; Tykwinski, R.R. *J. Am. Chem. Soc.* **2000**, *122*, 6917.

[73] Ohkita, M.; Ando, K.; Suzuki, Tsuji, T. *J. Org. Chem.* **2000**, *65*, 4385

orientated in the correct geometry by incorporating a Dewar benzene subunit into the precursor's structure. The angular nature of the Dewar benzene allowed for the successful dimerization of **113** to the novel cycle, **114**. Irradiation of **115** caused the aromatization of the Dewar benzene units and caused the acetylene linkages to bend with a bond angle of 168°.



Scheme 29. The synthesis of a strained phenylacetylene cyclophanes using a novel dewar benzene approach.

As the deformation from linearity increases it is expected that the reactivity of the alkyne will increase. Consequently, Oda and co-workers synthesized the symmetrical, nearly planar triyne **117**.[74] The strained alkynes displayed dienophilic character as exposure to excess cyclopentadiene resulted in double addition to give **118** (Scheme 30). The measured alkyne angle, α, in **118** was 159°. AM-1 calculations of enediyne **119**

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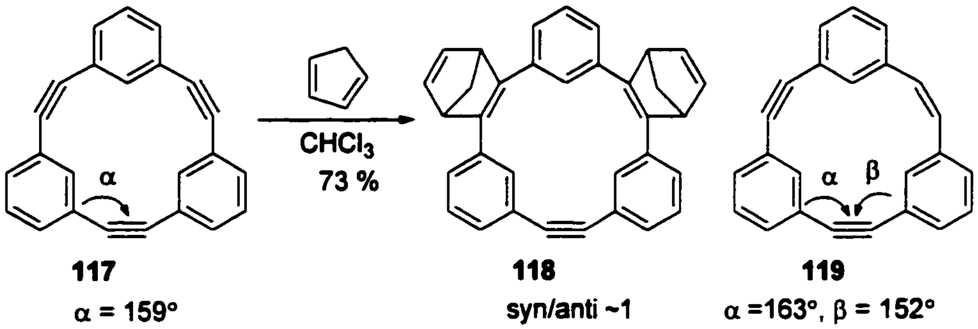
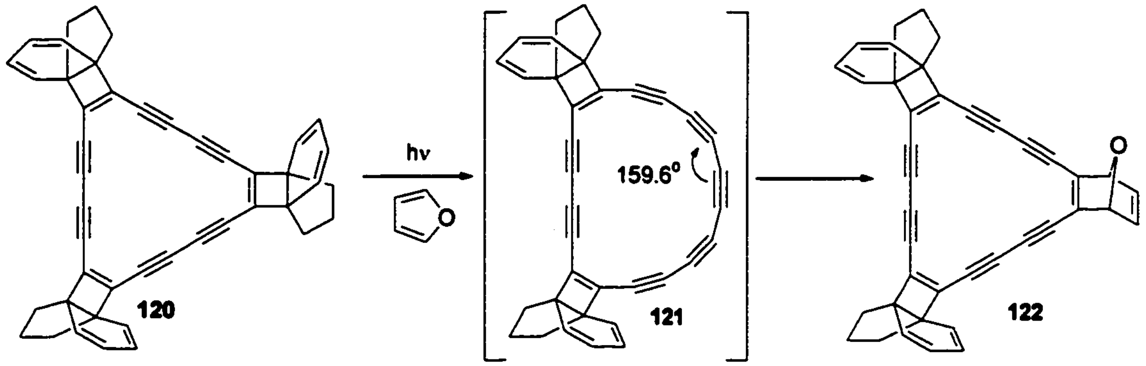
[74] Kawase, T.; Ueda, N.; Oda, M. *Tetrahedron Lett.* **1997**, *38*, 6681.

revealed that a significant degree of strain would still remain in the triple bonds (β = 152°) after an initial cycloaddition. Consequently, the second cycloaddition to afford **118** was observed, consistent with the sole formation of this diadduct.



Scheme 30. Strained alkyne linkages can exhibit dienophilic character and undergo Diels-Alder reaction.



Scheme 31. Generation of reactive polyynes by [2+2] cycloreversion and trapping with dienes.

Tobe and co-workers studied reactive polyynes generated by the [2+2] cycloreversion of [4.3.2]propellatriene derivatives.[51] In the process of studying cycloalkynes, they observed that they could trap the reactive polyyne from the cycloreversion of **120** in furan to generate the adduct **122**. The di- and tri-adducts were also observed during detailed NMR experiments. Computer AM-1 calculations suggested that an intermediate polyyne **121** would have a reactive central alkyne with a bent triple bond angle of approximately 159°.

CHAPTER 2: HEXASUBSTITUTED ENE-YNE CYCLOPHANES AS NOVEL PRECURSORS TO FULLERENES.

2.1 Cyclotrimerizations of Acetylenes *en route* to Hexasubstituted Benzenes.

Rubin's strategy towards the synthesis of fullerenes is based on the use of strained acetylenic cyclophanes.[40] His initial approach was to construct 1,3,5-trisubstituted acetylenic cyclophanes with the linkages between the benzene rings being composed of ene-yne units. Despite the success in synthesizing **64** and observing its coalescence to C_{60} in the gas phase, various attempts to transform the double bonds into triple bonds in solution failed.[52] Rubin postulated that the enediyne moieties present in **64** may undergo Bergman cyclization and start a reaction cascade leading to C_{60}. Incidentally, the π-orbitals of double bonds within enediynes are conjugated with those of the adjacent triple bonds, and these alkenes are often difficult to directly functionalize.[75] They, in turn, may be responsible for the inability of **64** to react in solution and form a highly strained cyclophyne.

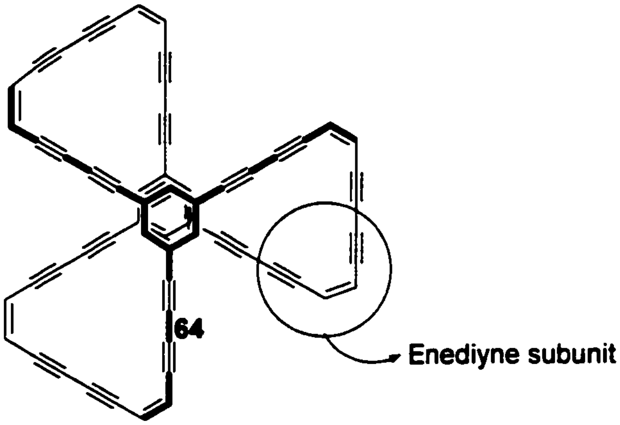


Figure 14. Rubin's basic design of 1,3,5-trisubstituted acetylenic cyclophanes containing enediyne moieties.

An alternate strategy may involve changing the sequence of the ene-yne linkages so that the double bond would be situated in the benzylic position. It was postulated that the reactive benzylic nature of such double bonds would allow them to undergo a bromination/dehydrobromination sequence for the installation of further acetylene units within the macrocycle.[75]

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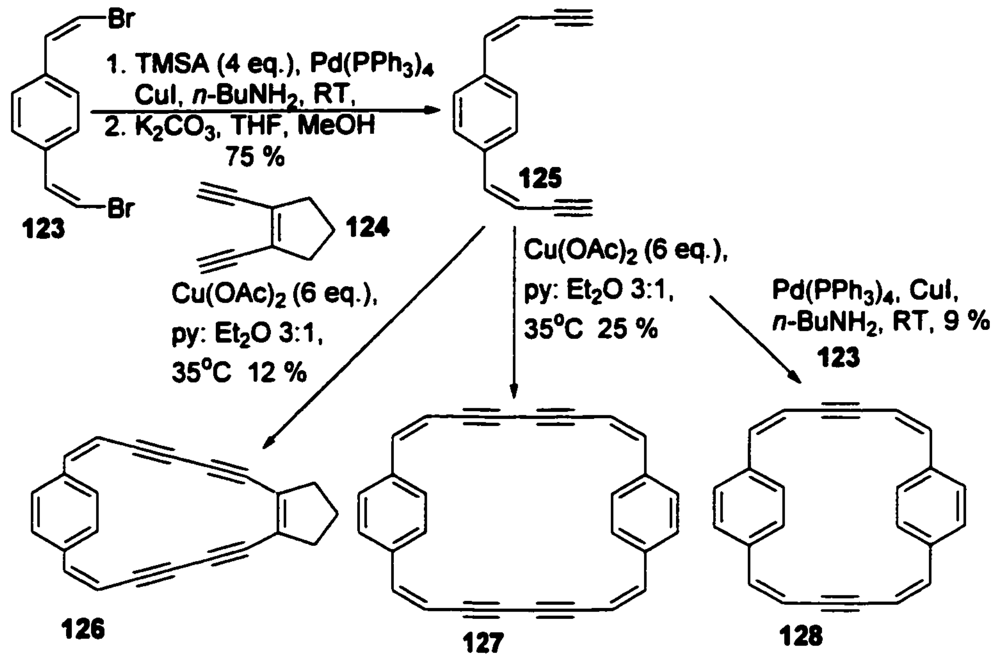
[75] Lu,Y.F.; Harwig, C.W.; Fallis, A.G. *Can J. Chem.* **1995**, *73*, 2253.



Scheme 32. Synthesis of various ene-yne cyclophanes dubbed "revolvenynes".

Previously, our laboratory had constructed a set of novel *para*-cyclophanes bearing ene-yne linkages (Scheme 32).[76] 1,4-Bis-(2-bromo-vinyl)-benzene, **123**, was the key intermediate in the synthesis of a number of cyclophanes constructed using palladium and copper based methodologies. Sonogashira coupling of **123** with two equivalents of TMSA installed the acetylene moieties for **125**. Subsequent desilylation and coupling with another equivalent of **123** produced the cyclophane, **128** as an orange solid. Dimerization of **125** using $Cu(OAc)_2$ in 3:1 pyridine: diethyl ether gave the dimer **127** as red needles. Unfortunately, attempts to collect X-ray crystallographic data have failed. Exposure of **123** to the identical copper-mediated dimerization conditions in the presence of **124** gave a mixture of the dimer and the cyclopentene cyclophane, **126**. The structure of **126** was confirmed by X-ray crystallography. The rotation of the central benzene rings in these cyclophanes were not restricted by the bridges. This contrasts cyclophanes bearing saturated linkages in which hindered rotation is observed. Free rotation of the benzene rings was still observed at temperatures as low as −60 °C by ^1H NMR measurements. In addition, computer modeling of these cyclophanes suggested that they should coil and adopt a helical twist. Calculations performed by Rubin and co-workers have suggested that similar acetylenic cyclophanes require little energy to racemize (~ 7.5

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[76] Romero, M.A.; Fallis, A.G. *Tetrahedron Lett.* **1994**, *35*, 4711.

kcal/mol).[52] Therefore, resolution of the individual enantiomers may require a chemical bias to force the molecule to remain in one conformation.

We envisioned that the benzylic ene-yne linkages would prove useful in the construction of fullerene-like precursors and cages. However, moving the double bond adjacent to the benzene ring necessitated the synthesis of hexasubstituted benzenes in order to obtain a cyclophane containing 60 carbon atoms. A retrosynthetic analysis is outlined in Figure 15. We chose to explore a cyclotrimerization of **129** as the key step in the formation of the intermediate 30 carbon hexayne, **130**. The cyclotrimerization reaction is expected to occur at the least sterically encumbered central alkyne. Dimerization of **130** forms the 60 carbon cyclophane, **131**, which may undergo transformation to C_{60} in the gas phase. A bromination/dehydrobromination strategy may also generate the strained cyclophyne, **132** in solution and collapse to buckminsterfullerene.

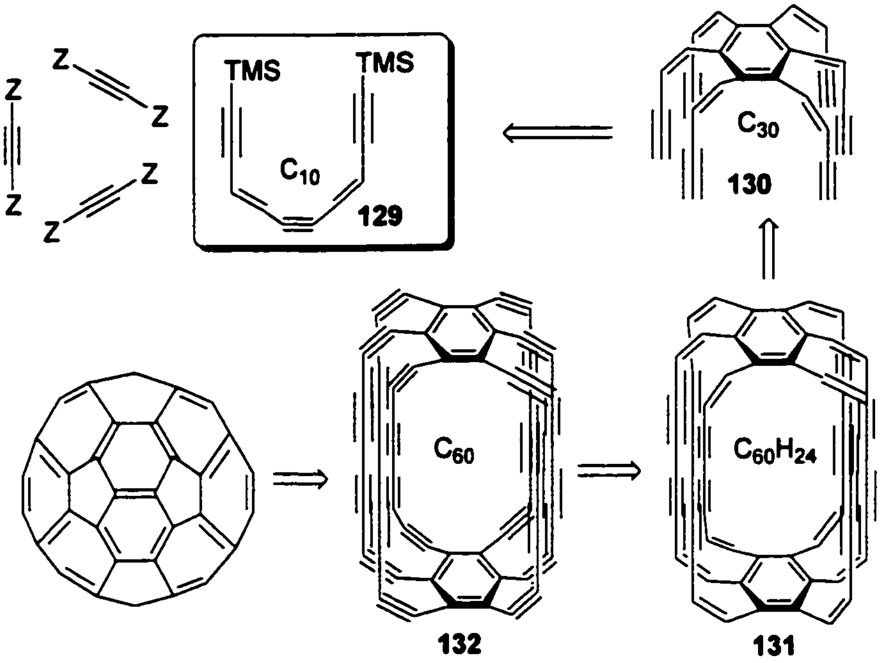
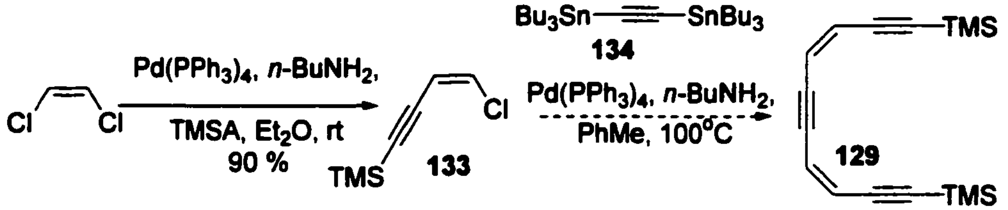


*Figure 15. Retrosynthetic analysis of C_{60} fullerene reveals the cyclotrimerization of **129** as the key step towards hexasubstituted acetylenic cyclophanes.*

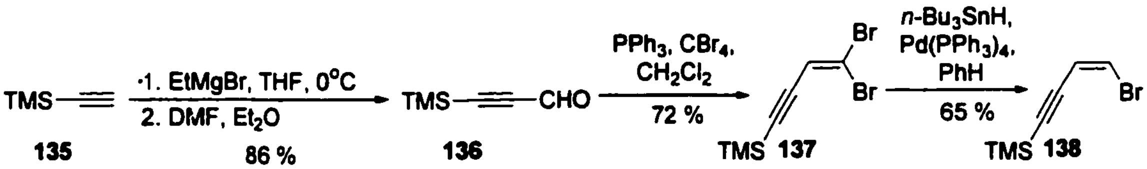
The synthesis of the key triyne unit, **129** is described in Scheme 33. The initial route made use of the reactivity of vinylchlorides. Commercially available *cis-*

dichloroethylene was reacted with 0.5 equivalents of TMSA to reduce any coupling.[77] The resuting ene-yne, **133** could then be coupled twice to the bis-(tri-*n*-butylstannyl)ethylene, **134**. Unfortunately, the Stille reaction did not occur and we turned to an alternate strategy using vinylbromides.



*Scheme 33. First generation synthesis of triyne, **129**, through coupling of vinyl chlorides.*

Trimethylsilylacetylene was deprotonated using ethylmagnesium bromide and the resulting anion trapped with DMF to give the aldehyde, **136**. A Corey-Fuchs reaction yielded dibromide, **137**, however, it was necessary to remove the *E*-bromide (external) atom.[78] We predicted that *Z*-bromide (internal) would be shielded due to steric hindrance and that it may be possible to remove the other halogen selectively. Exposure to tri-*n*-butyltinhydride and tetrakis(triphenylphosphine)palladium(0) gave the desired product in 65% yield. Only traces of dehalogenated species from replacement of both bromines by hydrogen were observed by TLC and GC MS analysis.



*Scheme 34. Synthesis of the bromo ene-yne, **138**.*

Acetylene **134** was synthesized by treating 1,1,2-trichloroethylene with 3 equivalents of *n*-butyllithium to generate the dilithiumacetylide and the dianion was quenched with tri-*n*-butylstannyl chloride (Scheme 35). Quenching with the more reactive but more toxic trimethylstannylchloride produced a volatile and difficult to handle clear liquid. Consequently, the *n*-butyl derivative underwent a Stille coupling with two equivalents of **138** to produce the desired triyne, **129**, in 73% yield.[77b]

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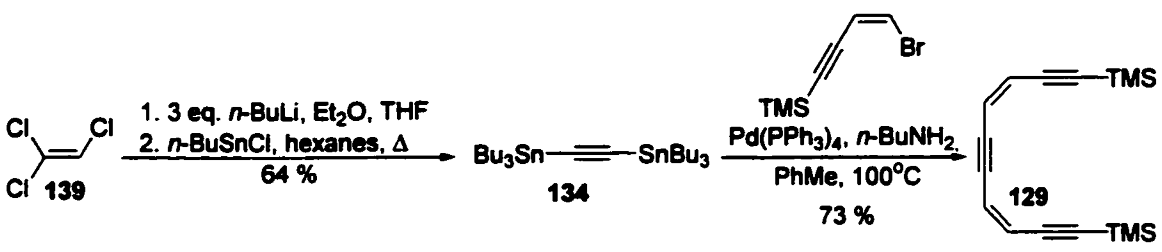
[77] (a) Lu, Y-F.; Harwig, C.W.; Fallis, A.G. *J. Org. Chem.* **1993**, *58*, 4202. (b) Manwell, J.; Fallis, A.G. *unpublished results.*

[78] Corey, E.J.; Fuchs, P.L. *Tetrahedron Lett.* **1972**, 3769.



Scheme 35. Synthesis of the triyne, 129.

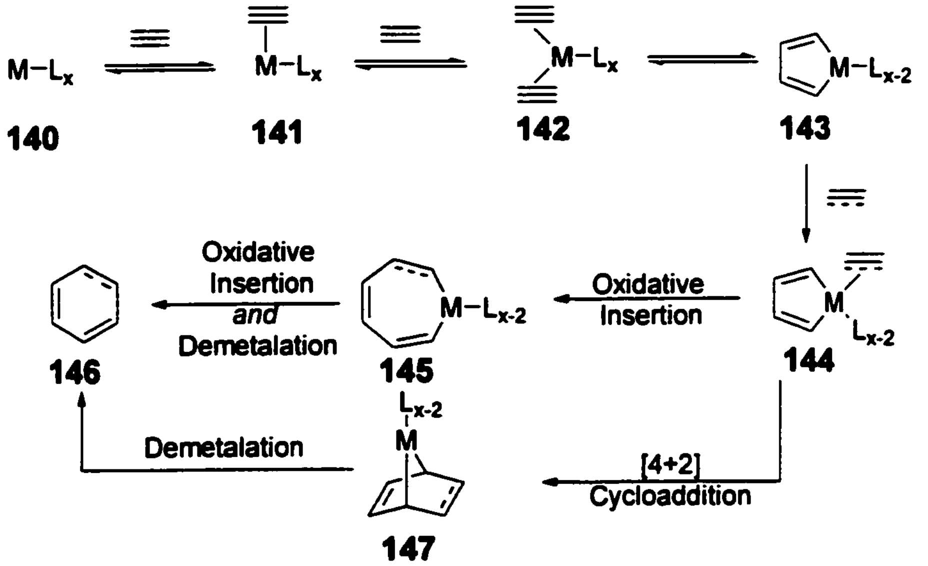


Figure 16. Possible mechanisms for the cyclotrimerization of two alkynes and a third unsaturated cycloaddend.

The cyclotrimerization of acetylenes provides a regiospecific method for building substituted benzenes and heteroaromatics.[79] Vollhardt's early studies of [2+2+2] cycloadditions with α,ω-diynes and monoalkynes showed that preorganization by $CpCo(CO)_2$ leads to successful transformations, despite strained synthetic intermediates. Low valent transition metal complexes best catalyze the reaction. Mechanistically, two alkyne moieties must complex before oxidative coupling may form the metallocyclopentadiene intermediate (Figure 16). Formation of this intermediate has been proposed as being responsible for the observed chemoselectivity. Co-ordination of the third acetylene produces an unisolatable synthetic intermediate through which two possible pathways may produce products. Entry of the alkyne moiety into the co-ordination sphere of the metal allows for insertion and formation of a metalloheptatriene,

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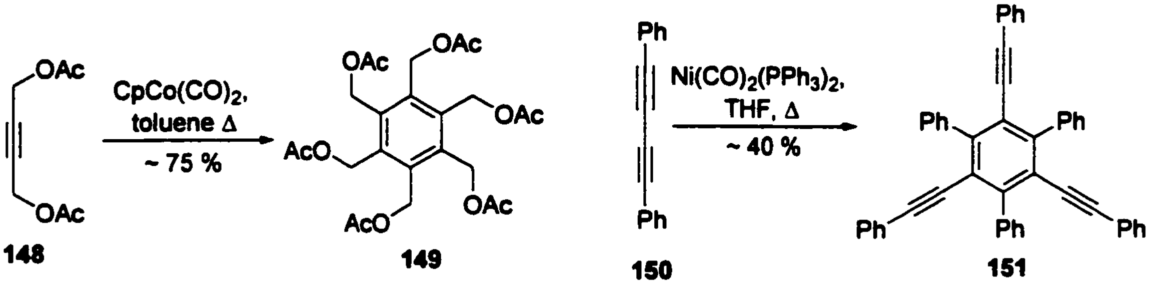
[79] Varela, J.A.; Castedo, L.; Saa, C. *J. Org. Chem.* **1997**, *62*, 4189.

followed by reductive elimination to give the product. A [4+2] Diels-Alder reaction can also result in the formation of the desired product and has been observed with electron poor dienophilic alkynes.[80]

The continuing importance of the [2+2+2] cycloaddition as a synthetic tool has fueled continual interest in the development of new catalyst systems with varying degrees of reactivity, including complexes of palladium,[81] nickel,[82] titanium,[83] iridium,[84] and ruthenium.[85] Despite the variety of catalyst systems available, the cobalt complexes [$CpCo(CO)_2$, $CpCo(PPh_3)_2$[86] and $CpCo(cod)$] and nickel complexes [$Ni(PPh_3)Cl_2$, $Ni(CO)_2(PPh_3)_2$] remain the most popular catalysts for the reaction (Scheme 36).[87]



Scheme 36. Cyclotrimerization of acetylenes with nickel and cobalt complexes.

The various attempts at the cyclotrimerization of **129** are outlined in Table 1. Cyclization in the presence of $Co_2(CO)_8$ in refluxing benzene disappointingly gave only decomposition of the starting material (entry 1). Exposure to $CpCo(CO)_2$ in refluxing toluene also failed to induce any reaction (entry 2). Due to the constant formation of decomposed starting material at elevated temperatures, trimerization was conducted at room temperature but no reaction occurred. Additionally, cyclization at room temperature with nickel catalyst also failed to give any reaction and increasing the temperature to reflux continued to decompose the triyne (entries 4 and 5 respectively). A palladium catalyst also failed to yield trimerization due to the instability of the starting

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[80] McAlister, D.R.; Bercaw, J.E.; Bergman, R.G. *J. Am. Chem. Soc.* **1977**, *99*, 1666.

[81] Brown, L.D.; Itoh, K.; Suzuki, H.; Hirai, K.; Ibers, J.A. *J. Am. Chem. Soc.* **1978**, *100*, 8232.

[82] (a) Ikeda, S.; Mori, N.; Sato, Y. *J. Am. Chem. Soc.* **1997**, *119*, 4779. (b) Ikeda, S.; Watanabe, H.; Sato, Y. *J. Org. Chem.* **1998**, *63*, 7026.

[83] (a) Hill, J.E.; Baliach, G.; Fanwick, P.E.; Rothwell, I.P. *Organometallics*, **1993**, *12*, 2911. (b) Johnson, E.S.; Balaich, G.J.; Rothwell, I.P. *J. Am. Chem. Soc.* **1997**, *119*, 7685.

[84] Takeuchi, R.; Tanaka, S.; Nakaya, Y. *Tetrahedron Lett.* **2001**, *42*, 2991.

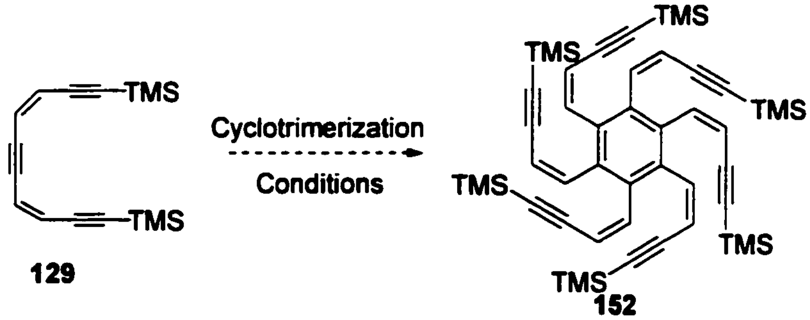
[85] Yamamoto, Y.; Kitahara, H.; Ogawa, R.; Itoh, K. *J. Org. Chem.* **1998**, *63*, 9610.

[86] Wakatsuki, Y.; Kuramitsu, T.; Yamazaki, H. *Tetrahedron Lett.* **1974**, *51*, 4549.

[87] Bonnemann, H. *Angew. Chem, Int. Ed. Engl.* **1985**, *24*, 248.

material (entry 6). Experimental studies revealed the triyne to be unstable above temperature of 40 °C.[77b] This observation made it difficult to imagine cyclotrimerization of the triyne since these reactions are typically conducted at high temperatures.

*Table 1. Attempts at producing the hexasubstituted precursor, **152**.*



Entry	Conditions	Results
1	$Co_2(CO)_8$, benzene, Δ	Decomposition
2	$CpCo(CO)_2$, toluene, Δ	Decomposition
3	$CpCo(CO)_2$, toluene, rt	No reaction
4	$Ni(PPh_3)_2Cl_2$, THF, rt	No reaction
5	$Ni(CO)_2(PPh_3)_2$, THF, Δ	Decomposition
6	$Pd(CH_3CN)_2Cl_2$, toluene, Δ	Decomposition

Subsequently, it was decided to investigate the cyclotrimerization of more stable acetylenes. The large number of conjugated acetylene units in **129** may have been unstable at high temperatures. Therefore the substrate was altered by removing the silyl protected triple bonds creating an acetylene bearing vinyl chlorides or bromides for further elaboration after the key step. (Figure 17).

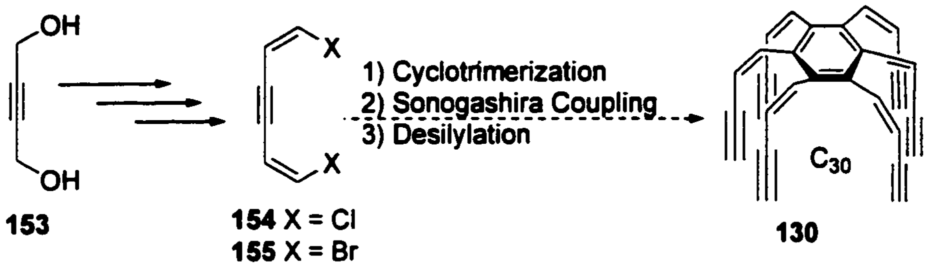
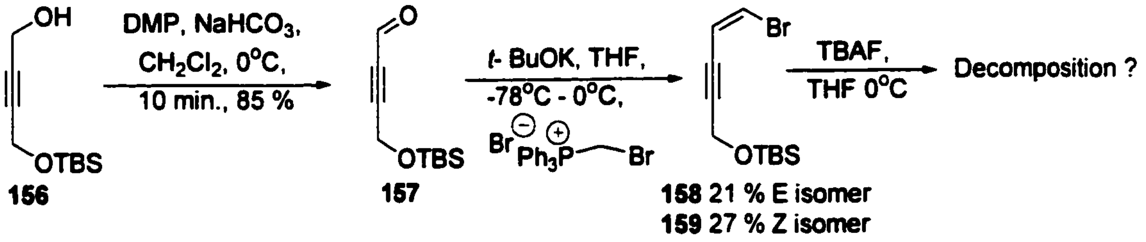


*Figure 17. An alternate substrate for the cyclotrimerization and synthesis of **130**.*

The synthetic sequence for **155** commenced with 1,4-butyndiol (Scheme 37). Oxidation of both hydroxyls to the but-2-yndial was not possible because of its instability above –90 °C. Therefore a sequential conversion of the diol to the dibromide was necessary. Monoprotection with TBSCl gave **156** allowing for oxidation of the remaining propargylic alcohol. Oxidation with PDC in DMF or CH_2Cl_2 produced small traces of aldehyde after 2 h. Extending the reaction time to 20 h was unsuccessful at producing any more than traces of **157**. Changing the oxidizing agent to the Dess-Martin periodinane in buffered dichloromethane provided the aldehyde, **157**, as a colourless oil in 85%. Wittig reaction of aldehydes with bromo- or chloromethyltriphenylphosphorane is known to give large product ratios favoring the *Z*-isomer.[88] Unfortunately, a Wittig reaction gave a combined 48% yield of the two vinyl bromide isomers. The desired *Z*-isomer was isolated in 27% yield. Although the Wittig reaction was not optimized, it was decided to continue towards the target, **155**. Deprotection of the silyl protected ether with acidic conditions was frowned upon because it was thought that isomerization of the double bond may occur. Consequently, fluoride ion was used to effect desilylation but upon addition of TBAF at 0 °C the reaction mixture turned red and continued to darken. Within minutes, the dark black solution showed no sign of any product or starting material by TLC analysis.



Scheme 37. Attempted synthesis of the dibromide, 155, via *Wittig reaction.*

Attempts to synthesize the vinyl chloride derivative **154** were similarly disappointing as described in Scheme 38. The known chloro ene-yne, **160**, was treated with TBAF to effect desilylation and the reaction mixture was subjected immediately to Sonogashira coupling conditions by adding the reaction mixture directly to a solution of

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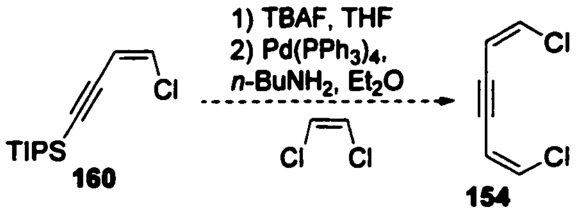
[88] Matsumoto, M.; Kuroda, K. *Tetrahedron Lett.* **1980**, *21*, 4021.

$Pd(PPh_3)_4$ and *n*-$BuNH_2$ in Et_2O. No desired product was observed, which may be due to the volatility of the deprotected intermediate.



Scheme 38. Sonogashira coupling strategy for the synthesis of ***154***.

2.2 Hexasubstituted Benzenes *via* Diels-Alder Reaction of Cyclopentadienones.

In revising our approach to hexasubstituted cyclophanes, we choose to investigate the possibility of constructing the C_{30} derivative, **130**, through the Diels-Alder reaction of an activated cyclopentadienone or the corresponding ketal, **162**, with the triyne, **129** (Figure 18). Four-fold Stille coupling of the known 2,3,4,5-tetrabromo-1,1-dimethoxycyclopentadiene, **33** with the stannylene-yne, **161** should give a protected, **162**. Following desilylation, **162** could then undergo Diels-Alder reaction or alternatively, the ketal could be hydrolyzed and the cyclopentadienone could then participate in the reaction.

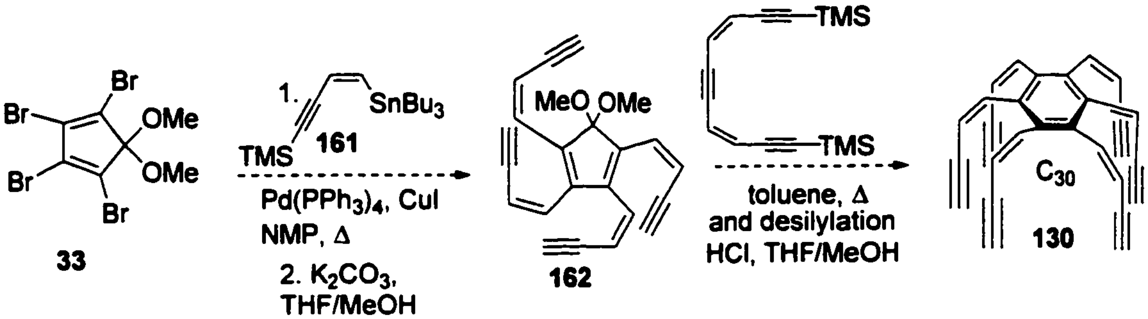


Figure 18. A Diels-Alder strategy for the synthesis of the C_{30} *precursor,* ***130***.

Previous studies on these systems for the synthesis of carbon networks revealed that the tetra-chloro analog, **163**, interestingly couples primarily at the 3- and 4- positions furthest from the ketal center. It was envisioned that this behavior could be taken advantage of to synthesize novel cyclophanes (Figure 19). If **163** underwent coupling at the 3- and 4- positions selectively, and was subjected to a Diels-Alder reaction with **129** (or another suitably functionalized acetylene that would allow for further installation of the ene-yne linkages), then it would be possible to gain access to the precursor, **165**. The geometric advantages of **165** over normal cyclophanes are two-fold: the bent bicyclic skeleton orients the two sets of linkages apart from each other and the two methoxy groups necessarily force the linkages onto the same side of the molecule. These geometric constraints are not present in the benzenoid analogs, where the "arms" or linkages may rotate freely in space. During copper-mediated dimerization, this may lead to the formation of unwanted cyclizations and isomers. Dimerization of **165** should give the cage structure, **166**. Treatment of **167** with acid should hydrolyze the ketal to the corresponding ketone and aromatization with loss of CO(g) should produce the aromatic cyclophane, **131**. The remaining ene-yne linkages could then be installed *via* cross-coupling chemistry, the silyl protecting groups removed and the remaining terminal acetylene groups of ene-yne bridges dimerized to give the target, **131**.

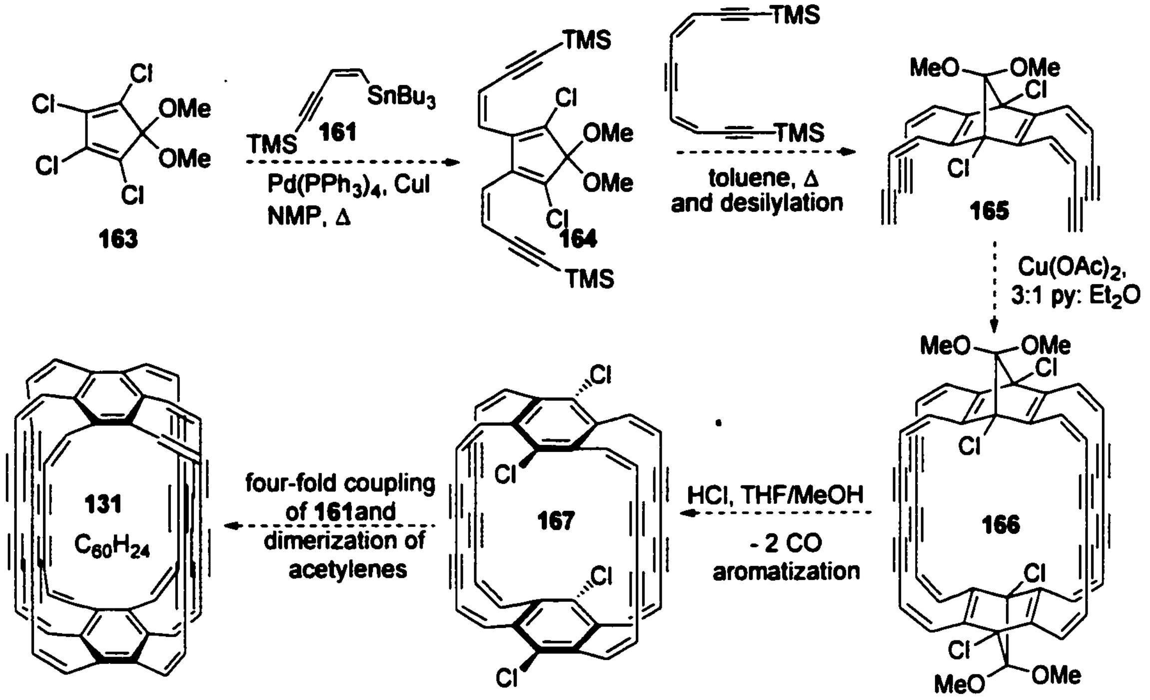
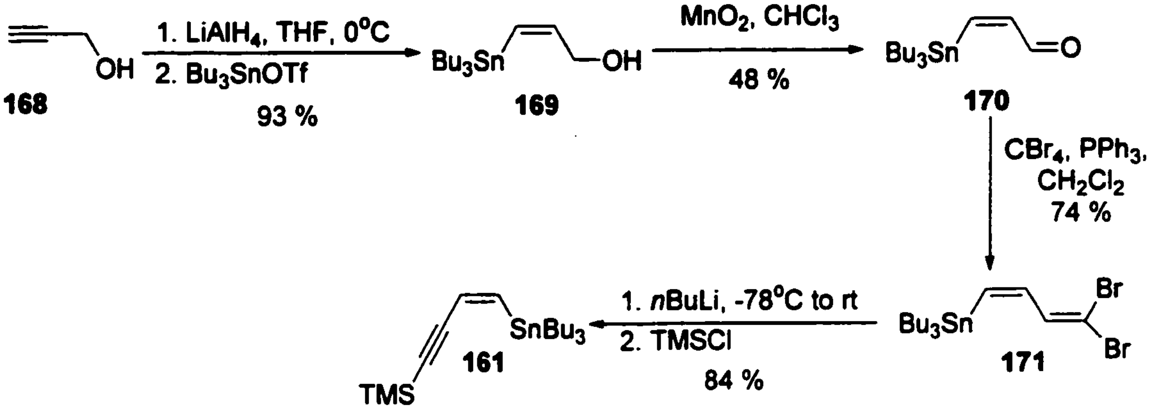


Figure 19. Formation of hexasubstituted cyclophanes using the Diels-Alder adducts of 2,3,4,5-tetrachloro-1,1-dimethoxycyclopentadiene.

Either of the above two synthetic strategies rely on the use of a suitable ene-yne coupling partner. The Stille reaction was investigated due to its mild reaction conditions and the stability of the organotin derivatives. Previously, our lab had investigated the synthesis of trimethyl-(4-tributylstannanyl-but-3-en-1-ynyl)-silane, **161** (Scheme 39).[77b] Starting from commercially available propargyl alcohol, hydroalumination of the triple bond followed by quenching of the organoaluminate with tri-*n*-butylstannyltriflate gave **169** in 93% yield. Curiously, quenching with the tri-*n*-butylstannylbromide or chloride resulted in no isolated product. Oxidation of the allylic alcohol with MnO_2 occurred in a modest 48% yield. The remaining starting material could be recovered and recycled to produce additional aldehyde. A Corey-Fuchs reaction was used to convert aldehyde, **170** to the corresponding acetylene. Treatment with CBr_4 and PPh_3 in CH_2Cl_2 gave the dibromoolefin in 74% yield. Treatment of **171**, with two equivalents of *n*-BuLi and quenching of the resulting acetylide with trimethylsilylchloride gave the desired product in 84% yield. This synthesis had several drawbacks in scaling up. The allylic alcohol, **169**, must be purified by chromatography, the cost of tri-*n*-butylstannyltriflate is very high, and the aldehyde, **170**, must be produced by recycling the alcohol over several cycles. Due to the large quantities of material needed for the synthesis, it was necessary to pursue a one step synthesis.



Scheme 39. Previous synthesis of the stannylene-yne, 161.

There are several existing methods for the synthesis of stannylated ene-ynes,[89] however, we were encouraged by studies of Magriotis and co-workers on the tandem Claisen and Bergman condensations for access to tetrahydronaphthalenes (Scheme 40).[90] They were able to synthesize enediynes in a single step using higher order cuprates.

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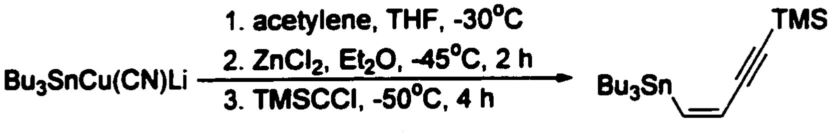
[89] (a) Marino, J.P.; Emonds, M.V.M.; Stengel, P.J.; Oliveira, A.R.M.; Simonelli, F.; Ferreira, J.T.B. *Tetrahedron Lett.* **1992**, *33*, 49. (b) Bujard, M.; Ferri, F.; Alami, M. *Tetrahedron Lett.* **1998**, *39*, 4243. (c) Trost, B.M.; Hachiya, I.; McIntosh, M.C. *Tetrahedron Lett.* **1998**, *39*, 6445. (d) Shirakawa, E.; Yosida, H.; Kurahashi, T.; Nakao, Y.; Hiyama, T. *J. Am. Chem. Soc.* **1998**, *120*, 2975.

[90] Magriotis, P.A.; Scott, M.E.; Kim, K.D. *Tetrahedron Lett.* **1991**, *32*, 6085.



60 % **161**

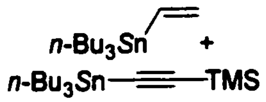
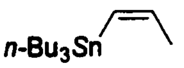
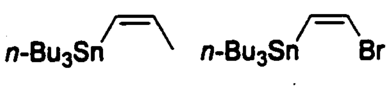
Scheme 40. Magriotis' use of higher order stannylcuprates for the synthesis of ene-ynes.

Unfortunately, our attempts to duplicate the higher order stannylcuprate technology failed in all instances (Table 2).

Table 2. Higher order stannylcuprates as intermediates in the synthesis of ene-yne products.

$$n\text{-Bu}_3\text{SnH} \xrightarrow[\text{2. CuCN, DMS, THF, -45°C} \;\; \text{3. -45°C to -20°C}]{\text{1. LDA, THF, -78°C}} [n\text{-Bu}_3\text{Sn(Me)CuCN]Li}_2$$

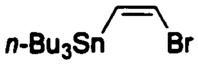
172

Entry	Additional Reagents	Products
1	1. Acetylene (g) 2. ZnCl$_2$ 3. TMSCCBr	
2	1. Acetylene (g) 2. ZnCl$_2$ 3. TMSCCI	Decomposition
3	1. Acetylene (g) 2. TMSCCI	No products observed
4	1. Acetylene (g) 2. ZnCl$_2$ 3. PhCCBr	No products observed
5	1. Acetylene (g) 2. MeI	
6	1. Acetylene (g) 2. ZnCl$_2$ 3. Br$_2$	

Repeating the published conditions with the trimethylsilylacetylenebromide gave traces of the vinyl-tri-*n*-butyltin and 1-(tri-*n*-butylstannyl)-2-(trimethylsilyl)ethylene as by-products (entry 1). These products were normally observed in these types of reaction as trace impurities. Altering the substrate to the more reactive iodide gave a product that decomposed upon isolation and could not be properly identified. Our results concur with Magriotis' reports that improved yields are obtained by the addition of a transmetalation step with $ZnCl_2$; omission of $ZnCl_2$ from the reaction mixture produced no product (entry 3). Attempts at investigating a model system using trimethylsilylphenylacetylene also failed to produce any ene-yne products (entry 4). The initial cuprate forming step was verified by quenching the reaction mixture with methyliodide and getting quantitative formation of 1-(tri-n-butylstannyl)-1-propene (entry 5). However, the quenching of the advanced zinc intermediate with elemental bromine was problematic and produced the vinylbromide and the unexplained formation of 1-(tri-n-butylstannyl)-1-propene (entry 6).

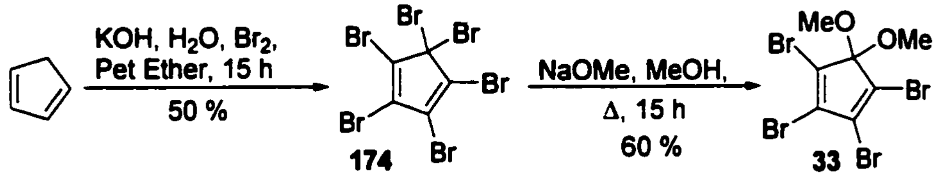
Table 3. Coupling reactions using an alternative preparation of the stannyl cuprate, 173.

$$(n\text{-}Bu_3Sn)_2 \xrightarrow[\text{3. CuCN, -45°C to -20°C}]{\text{1. } n\text{-BuLi, THF, 0°C} \quad \text{2. MeLi, THF, -78°C}} \underset{\mathbf{173}}{n\text{-}Bu_3SnCu(CN)Li}$$

Entry	Additional Reagents	Products
1	1. Acetylene (g) 2. $ZnCl_2$ 3. TMSCCBr	No products observed
2	1. Acetylene (g) 2. $ZnCl_2$ 3. TMSCCI	No products observed
3	1. Acetylene (g) 2. $ZnCl_2$ 3. PhCCBr	No products observed
4	1. Acetylene (g) 2. $ZnCl_2$ 3. Br_2	

The nature of the stannylcuprate was altered to investigate whether the reaction conditions could be improved; however, all of these attempts failed as well. Generation of the stannylacetylene complex, often described as a dianion equivalent, was accomplished by treatment of bis(tri-*n*-butylstannane) with *n*-BuLi, MeLi, and CuCN following a published procedure.[88] This organometallic intermediate also failed to couple with silyl-protected acetylene bromides and iodides (entries 1 and 2 respectively). Although coupling of the model phenylacetylene system failed to give any coupling products (entry 3), quenching of the zincated cuprate with bromine did produce the *cis*-1-(tri-*n*-butylstannyl)-2-bromoethene quantitatively.

Synthesis of the required cyclopentadiene precursors was accomplished following literature procedures. Exhaustive bromination of cyclopentadiene was accomplished using a literature procedure to give **174**.[91] However, following the literature guidelines with workup after 10 minutes failed to give any noticeable product. Extending the reaction time to 2 or 4 h and doubling the concentration of KOH/Br_2 also had no effect. Fortunately, vigorous stirring for 15 h caused the formation of a yellow precipitate which could be isolated and recrystallized from pentane to give brown cubic crystals. Submitting these crystals to a freshly prepared solution of sodium methoxide generated from sodium metal and methanol gave the ketal after isolation by pouring the reaction mixture onto ice. Filtration gave **33** in yields of 60-65%.[92]



Scheme 41. Literature synthesis of the tetrabromocyclopentadiene, 33.

Diels-Alder studies at low temperatures with unactivated dienophiles were initiated with commercially available tetrachloro derivative, **163** (Table 4).[93] A [4+2] cycloaddition with diiodoacetylene would install extra halides for cross-coupling reactions. Unfortunately, no reaction occurred at room temperature and heating to reflux in toluene also failed and the starting materials were recovered (entries 1 and 2). Cycloaddition with 1,4-butandiol would install hydroxyl groups that could be oxidized to aldehydes and then converted to acetylenes or to bromoalkenes using Wittig chemistry. Again, no reaction occurred at room temperature and heating in THF failed to initiate any reaction (entry 3). Turning to activated acetylenic dienophiles, treating **163** with

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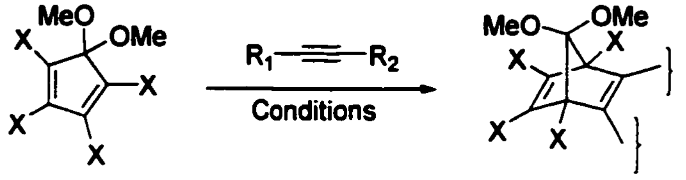
[91] West, R.; Kwitowski, P.T. *J. Am. Chem. Soc.* **1968**, *90*, 4697.

[92] Pews, R.G.; Roberts, C.W.; Hand, C.R. *Tetrahedron* **1970**, *26*, 1711.

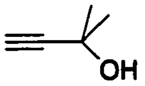
[93] Singleton, D.A.; Merrigan, S.R.; Thomas, A.A. *Tetrahedron Lett.* **1999**, *40*, 639.

dimethylacetylene diester (DMAD) at -78 °C with a Lewis acid catalysis (entry 4) gave traces of the product. Raising the temperature to ambient temperature did not produce any increase in yield. Similarly, refluxing in THF only served to decompose the starting materials.

*Table 4. Diels-Alder studies of acetylenic dienophiles with **163** and **33**.*



163 R = Cl
33 R = Br

Entry	X	R_1	R_2	Conditions	Product(s)
1	Cl	I	I	toluene, Δ	Recovered Starting Material
2	Cl	I	I	toluene, rt	Recovered Starting Material
3	Cl	CH_2OH	CH_2OH	THF, Δ	Recovered Starting Material
4	Cl	CO_2Me	CO_2Me	Me_2AlCl, CH_2Cl_2, -78 °C	trace
5	Cl	CO_2Me	CO_2Me	Me_2AlCl, CH_2Cl_2, rt	trace
6	Cl	CO_2Me	CO_2Me	THF, Δ	Recovered Starting Material
7	Br	I	I	toluene, Δ	Recovered Starting Material
8	Br	TIPS	 OH	xylene, Δ	Recovered Starting Material
9	Br	CO_2Me	CO_2Me	benzene, Δ	Recovered Starting Material

Diels-Alder studies with the tetrabromo derivative, **33**, gave similar results to those obtained with the chloride derivative. Cycloaddition with diiodoacetylene, cycloaddition with 1,3-butadiynes, similar to those employed by Rubin and Tobe,[27,28] and heating with DMAD in benzene all continued to just give recovered starting material.

However, an interesting reaction occurred when **33** and DMAD were heated neat at 160 °C overnight. A single product was obtained and isolated by chromatography. The ^{1}H NMR spectrum showed two signals at δ 3.91 and 3.83 ppm and the former was twice the intensity of the latter. The ^{1}H NMR spectrum would tend to point to the product being the symmetrical compound, **176** (Figure 20). This product could arise from Diels-Alder reaction with DMAD, followed a retro-[4+2] with loss of dibromoacetylene. The resulting cyclopentadiene intermediate would then undergo another Diels-Alder addition to give, **176**. The two methyl groups of the ester would account for the signal at 3.91 ppm while the methoxy group signal would be half the intensity (accounting for three hydrogens). However, the ^{13}C NMR data contradicts that interpretation of the ^{1}H NMR results. The number of signals in the ^{13}C spectra pointed towards the product, **175** arising from a single cycloaddition, however, it also showed the existence of two different carbonyl groups. Mass spectrometric analysis did not produce the M$^+$ signal of either of the two products. The structure of the product was solved by X-ray crystallography and was in agreement with the spectral evidence obtained.

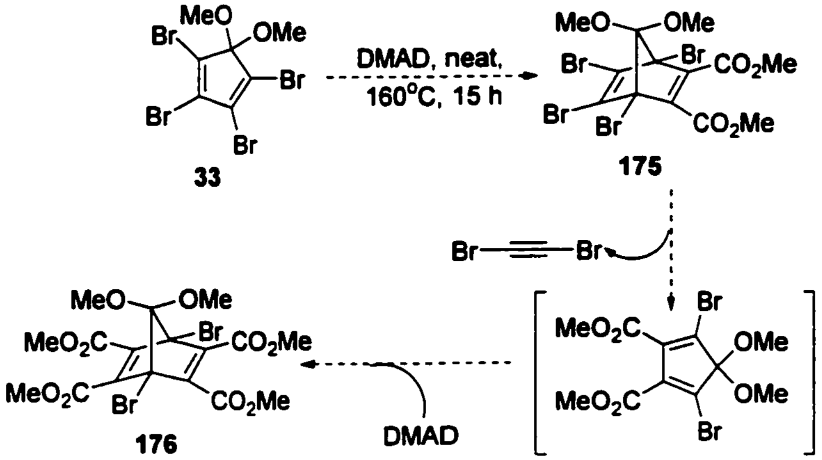
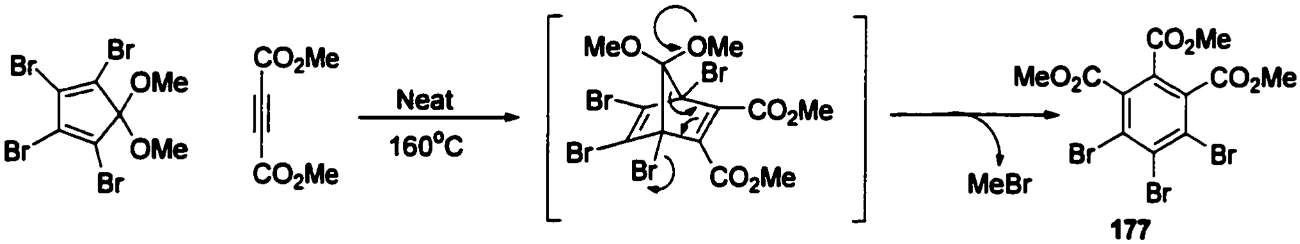


Figure 20. Possible products formed from the Diels-Alder reaction of 33 with DMAD.

The product was recrystallized as colourless needles by slow cooling of a hot methanol solution. The X-ray crystal structure revealed the formation of an aromatic product bearing three bromine atoms and three methyl ester groups. A plausible mechanism for the formation of this product is outlined in Figure 21. The lone pair of electrons from a methoxy group could form the third methyl ester group and consequently causes the breaking of the bicyclic skeleton, expulsion of a bromide ion and aromatization.



177

Figure 21. Plausible mechanism for the formation of 177.

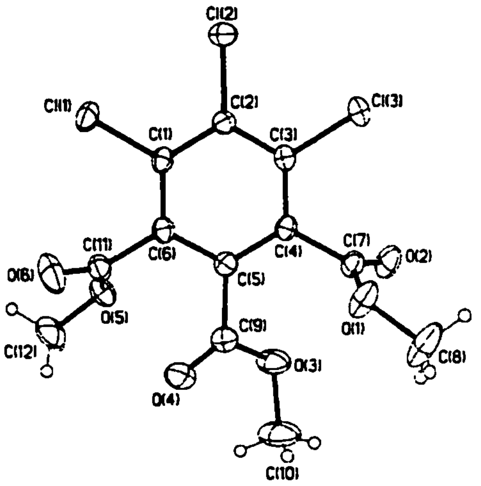


Figure 22. X-ray crystallographic analysis of the product, 177 revealed an aromatic product had formed.

Attempted Stille coupling of the stannylene-yne, **161**, with **33** failed after refluxing for 15 h. Altering the number of equivalents of the coupling partner, lengthening the reaction time, and changing the palladium catalyst from $Pd(PPh_3)_2Cl_2$ to $Pd(PPh_3)_4$ also had no effect on the reaction and the starting materials were recovered.

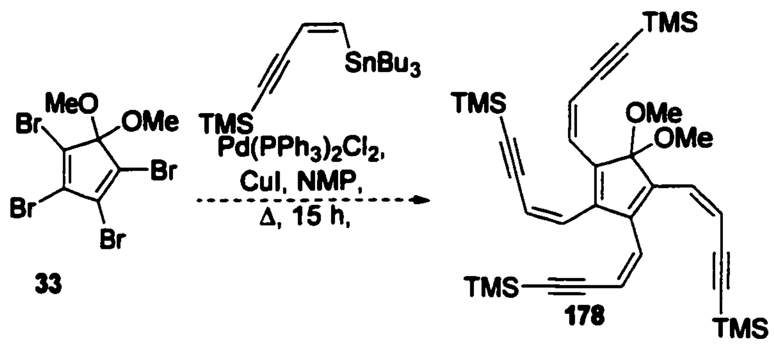
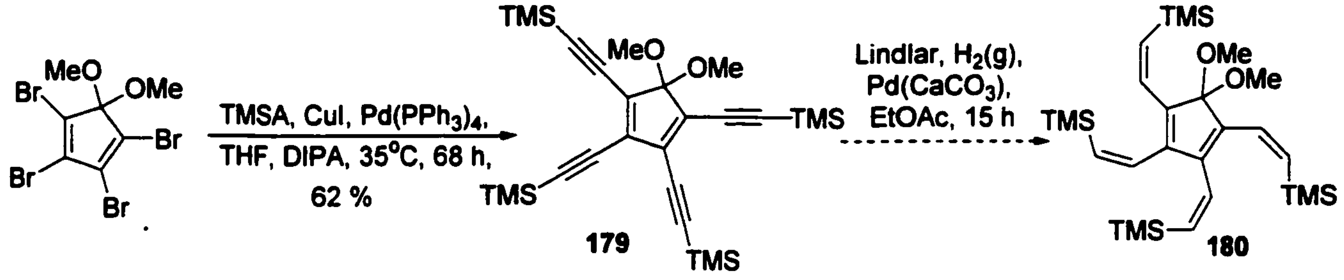


Figure 23. Attempted Stille coupling of 33 to produce 178.

The molecule, **180**, could be produced by a Lindlar reduction of **179**. The TMS-protecting groups could then be replaced by halogens and a second cross-coupling to install the necessary acetylene linkages. Following the protocol outlined by Tobe, four equivalents of TMSA were cross-coupled to **33** in 62% (Scheme 42).[27] Lindlar reduction of **179** using Pd(CaCO)$_3$ in ethyl acetate was unsuccessful in reducing the conjugated acetylenes and the starting material was recovered.



Scheme 42. Attempted Lindlar reduction of 179.

Conditions for the *in situ* generation of the Grignard reagents and subsequent palladium cross-coupling have been studied.[94] The Grignard **181** was formed and Pd cross-coupling failed to produce the tetra-substituted derivative, **182** or the bis-coupled, **183** (Scheme 43).

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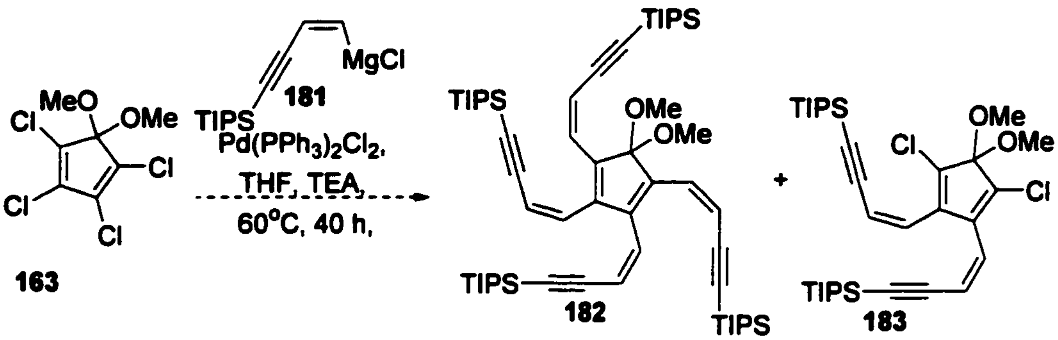
[94] (a) Ramiandrasoa, P.; Brehon, B.; Thivet, A.; Alami, M.; Cahiez, G. *Tetrahedron Lett.* **1997**, *38*, 2447.
(b) Reddy, N.P.; Tanaka, M. *Tetrahedron Lett.* **1997**, *38*, 4807.



*Scheme 43. Attempt at Grignard cross-coupling with the tetrachloro-**163**.*

CHAPTER 3: DESIGN AND SYNTHESIS OF NOVEL FAMILY OF PHENYL ACETYLENE CYCLOPHANES.

3.1 Design and Synthesis of Model Phenyl-yne Cyclophanes.

The synthetic applications for phenyl-yne linked acetylenic cyclophanes continue to expand. They form the basic building blocks for the construction of novel DBAs that are studied for their electronic behavior,[20a] propensity for aromaticity,[21] and as macrocyclic segments or mimics of novel acetylenic carbon networks.[18,25] They are also the major structural unit for forming 3-dimensional nanostructures,[34,35] with potential as fullerene precursors.[47a] Although phenyl-yne bridged cyclophanes have the potential to form well-defined and complicated structures, simple phenyl-yne macrocycles can be pyrolyzed at temperatures ranging from 200 °C to 800 °C to form carbon nanotubes and carbon onions.[48,49] They are also important fragments in the syntheses of bucky bowls, or bucky bits and "fullerenoids".[59,68]

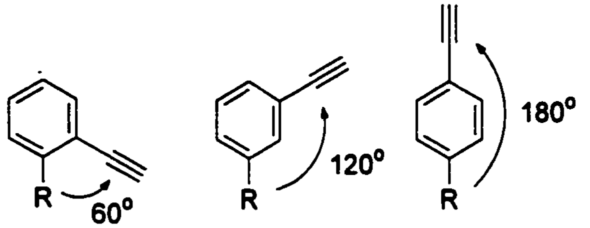


Figure 24. Various trigonal geometries attainable by phenyl-yne linkages.

The phenyl-acetylene bridges can be used to construct a wide variety of nanostructures or cyclophanes with different geometries due to the three trigonal angles at which the acetylene spacer can be orientated. Changing the position of the acetylene on the benzene with respect to other substituents alters the shape of the macrocycle (Figure 24). For example, changing a linkage from *ortho* to *meta* changes the angle from 60° to 120°.

Synthetically, the C_{sp}-C_{sp^2} bond is often installed using Sonogashira cross-coupling chemistry, however, the C_{sp^2}-C_{sp^2} bond between two phenyl groups can be achieved using a variety of coupling reagents. The reactivity of the organometallic component can be varied by changing the metal component from ZnBr (Negishi coupling), $SnBu_3$ (Stille coupling), MgBr (Grignard) or $B(OH)_2$ (Suzuki coupling). In addition to the diversity and availability of these metal-aryl species, their relative stability and the elimination of *cis-trans* isomerization problems encountered with ene-yne linkages, all contribute to making these linkages and cyclophanes attractive.

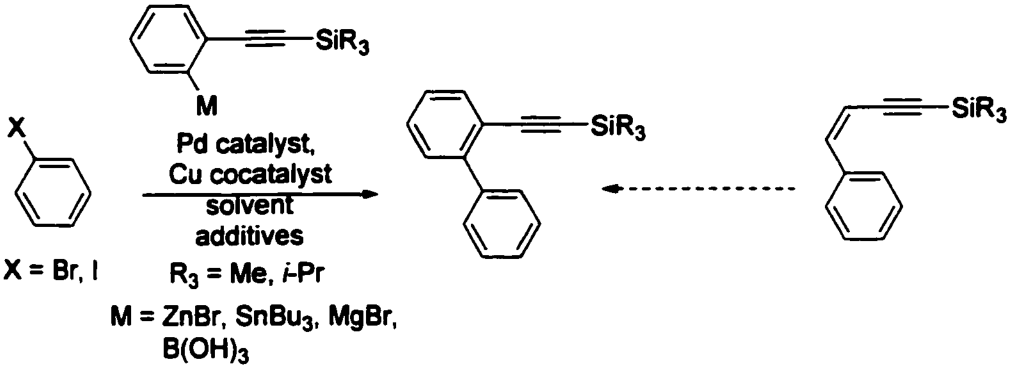
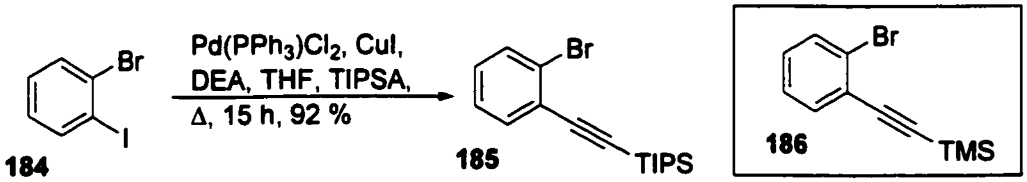


Figure 25. Various organometallic varieties of aryl coupling partners.

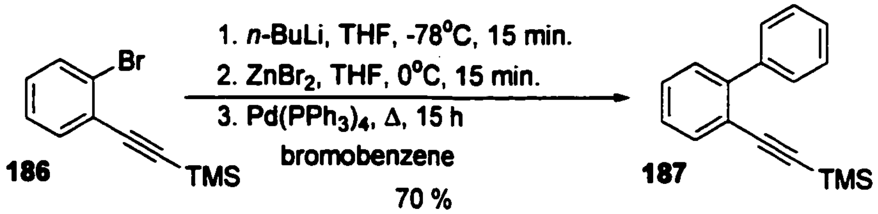
It was decided to investigate the synthesis of *para*-cyclophanes similar to the ene-yne species that were created previously. The following investigation represents studies into novel ring systems and have potential to act as precursors for fullerenes and as novel liquid crystals. Their synthesis would proceed using general methods and strategies that could be repeated by others.

The two different trialkylsilyl protected precursors, **185** and **186** were the key synthetic building blocks. Although the trimethylsilyl (TMS) protected derivative **186** is commercially available, the triisopropylsilyl protected, **185** required construction (Scheme 44). Sonogashira coupling of triisopropylsilylacetylene (TIPSA) in the presence of $Pd(PPh_3)_2Cl_2$ and CuI to 1,2-dibromobenzene with triethylamine only gave a 1:1 mixture of product and starting material. Furthermore, the two substances could only be separated by time-consuming preparative HPLC using a size-exclusive column. Changing the base to DEA also produced the identical ratio of product to starting material and continually adding fresh portions of TIPSA to the reaction mixture also failed in driving the reaction to completion. Using commercially available 2-bromo-1-iodobenzene, a selective Sonogashira coupling of one equivalent of TIPSA to the iodide at reflux for 15 h gave **185** in excellent 92% yield.[52]



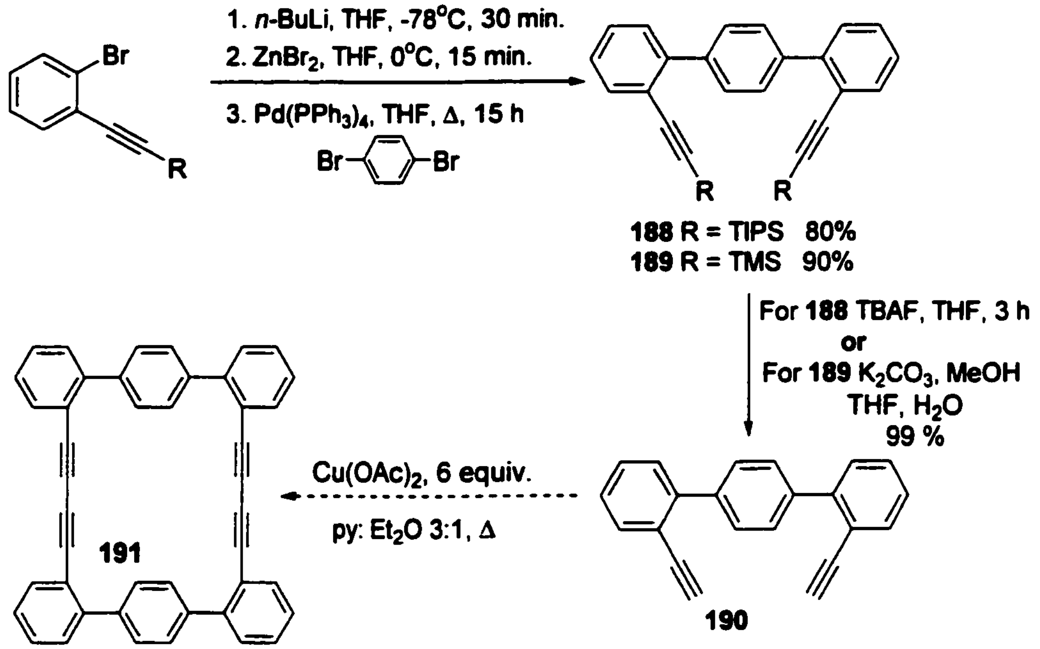
Scheme 44. Synthesis of TIPS and TMS protected building blocks.

A Negishi coupling protocol was adopted for the cross-coupling of phenyl groups. Traditional Stille coupling would require preparation of the tri-*n*-butylstannyl organometallic reagent prior to coupling, and after reaction, purification is complicated by co-elution of the nonpolar products with remaining tri-*n*-butylstannylhalides. The synthetic advantages to using a ZnBr-mediated coupling are that the organometallic reagent is generated *in-situ* by lithium-halogen exchange and transmetalation with $ZnBr_2$. Moreover, after the reaction the remaining zinc salts could easily be separated by chromatography or by organic/aqueous extractions. Transmetallation of **186** using *s*-BuLi after 15, 30 and 60 minutes showed incomplete formation of the organo-lithium. Instead, lithium-halogen exchange was complete after 10 minutes following treatment with *n*-BuLi. Transmetallation with $ZnBr_2$ in THF at 0 °C followed by warming to ambient temperature and addition of $Pd(PPh_3)_4$ and bromobenzene produced **187** after 15 hours at reflux in 70% yield (Scheme 45).



*Scheme 45. Negishi coupling of **186** to aryl substrates.*

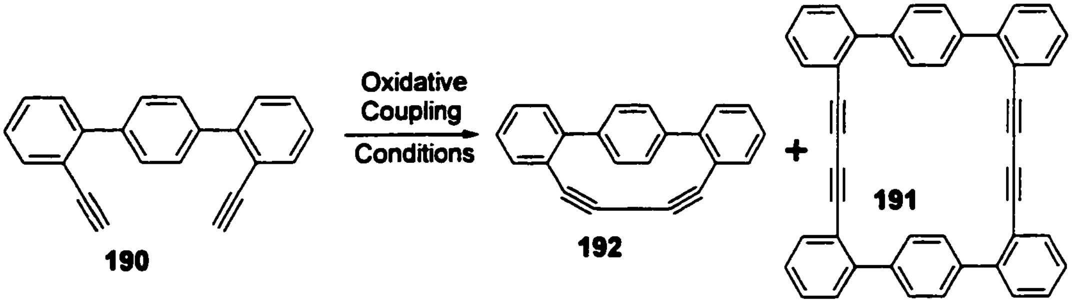
With optimized conditions in hand, it was decided to investigate the dimer **191** as a model system. Generating the organozincate of **185** or **186** followed by palladium catalyzed cross-coupling with 1,4-dibromobenzene gave the TIPS protected product, **188** in 80% yield and the corresponding TMS protected product, **189** in 90% yield (Scheme 46). Both of these compounds were deprotected in quantitative yield. The TIPS protecting groups were removed by treatment with TBAF in THF while the less hindered TMS groups were removed under basic conditions with potassium carbonate in MeOH and THF. Diacetylene, **190**, was then added to the reaction vessel by syringe pump under pseudo high dilution conditions to minimize polymerization of the starting material. After addition to a refluxing solution of $Cu(OAc)_2$ in 3:1 pyridine: diethyl ether a single product was formed. Surprisingly, the product isolated was the strained compound, **192** as an off-white solid in 90% yield (Table 5, entry 1)! The product **192** is the result of intramolecular reaction of the two terminal acetylenes.



Scheme 46. Design and synthesis of the model phenyl acetylene cyclophane, 191.

Attempts were made to alter the reaction conditions to see if it were possible to induce dimer formation. Lowering the reaction temperature produced two products. The dimer, **191** was isolated as a yellow solid that darkened over time in approximately 20% yield, however, the monomer was still the major product and was isolated in 50% yield. The reaction mixture turned from sapphire blue to an emerald green a few minutes after initial addition of the acetylene. However, protecting the solution from light by covering the round bottom flask with aluminum foil caused the reaction mixture to maintain the sapphire blue colour. Despite no colour change, the reactions gave similar product ratios (entries 2 and 3 respectively). Extending the reaction time resulted in decomposition of both products, although the monomer did degrade faster than the dimer product (entry 4). This may be due to the inherent strain present in the butadiyne bridge of the molecule. Hay type dimerization conditions disappointingly gave a mixture of products from which the dimer was isolated in less than 10% yield (entry 5)

*Table 5. Copper-mediated oxidative dimerization of **190**.*



Entry	Conditions	Yield (%) **192**	Yield (%) **191**
1	Cu(OAc)$_2$, 6 eq. py: ether 3:1, Δ, 3 h	90	-
2	Cu(OAc)$_2$, 6 eq. py: ether 3:1, rt, 3 h	43-58	18-26
3	Cu(OAc)$_2$, 6 eq. py: ether 3:1, rt, 3 h (protected from light)	54	17-21
4	Cu(OAc)$_2$, 6 eq. py: ether 3:1, rt, 2 d	10	17
5	CuCl, TMEDA, O$_2$, benzene, 3 d	-	<10

In order to establish the absolute structure of **191**, an X-ray crystallographic analysis was performed.[95] Crystals were grown from a variety of solutions employing chlorinated solvents as Rubin and co-workers have shown that halogenated solvents aid in the crystallization of similar hydrocarbons. Slow evaporation of a dichloromethane solution produced small clear needle-like crystals. Two stereoviews of the resulting structure are illustrated in Figure 26. The molecule adopts a twisted helical conformation as expected due the nature and rigidity of the phenyl-yne linkages. The central benzene rings are overlapping but are slightly offset from each other. The twisting occurs to a degree that one of the aromatic rings in the "arms" of the molecule may π-stack with one another. The molecule's "arms" extend outward to form a small cavity with a pincer-like arrangement that can trap a molecule of dichloromethane.[96]

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[95] Collins, S.K.; Yap, G.P.A.; Fallis, A.G. *Angew. Chem. Int. Ed.* **2000**, *39*, 385.

[96] Kochi J.K. *Angew. Chem. Int. Ed.*, **1998**, *37*, 1585.

This is especially evident in the crystal packing diagram of **191** as several molecules of dichloromethane are captured within the lattice, in a ~ 2:1 ratio (Figure 27).

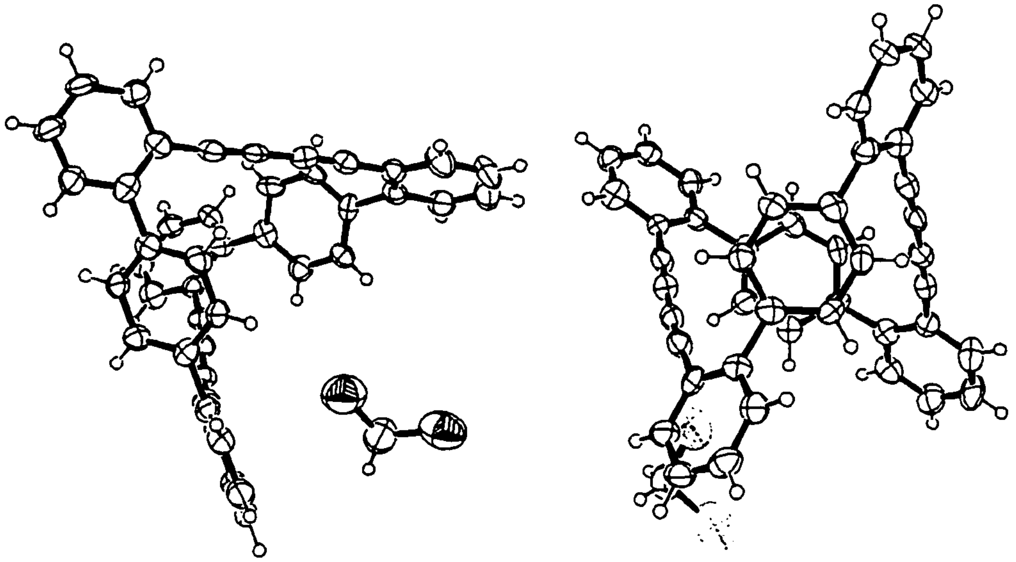


Figure 26. X-ray crystal structure of ***191*** *shows co-crystallization of dichloromethane. Views from the side (left) and the top (right).*

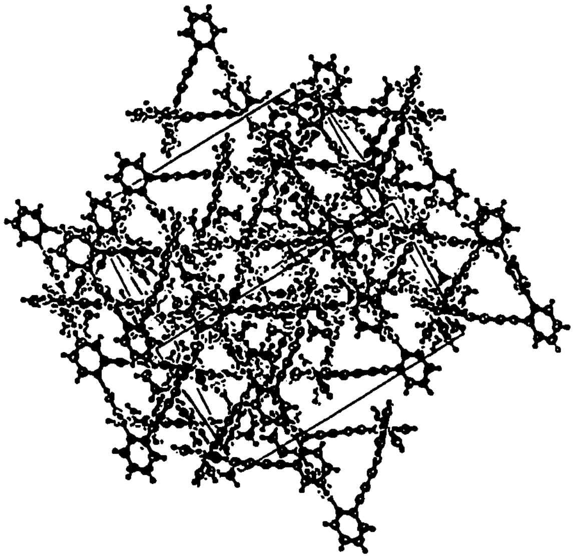
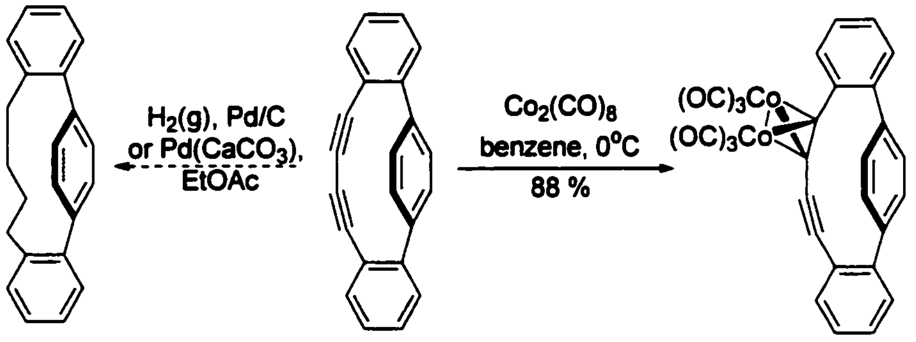


Figure 27. Crystal packing diagram of dimer ***191*** *displays intermittent crystallization of dichloromethane within the lattice.*

Despite the success in obtaining a crystal structure of the dimer, getting an X-ray crystal structure of the monomer was not as easy. The monomer was stable in the solid state but decomposed slowly over a period of weeks in mildly acidic solutions such as dichloromethane. Attempts to obtain more crystalline samples through chemical modification became necessary (Scheme 47). Although modification of the butadiyne bridge would make it difficult to obtain any interesting structural information about the strained linkage, a cobalt carbonyl derivative of the monomer, **192**, was synthesized.[97] When **192** was treated with dicobalt octacarbonyl in benzene at 2 °C, conversion to **194** was complete in 10 minutes. Unfortunately, **194** did not produce X-ray crystallographic quality crystals. Hydrogenation of the butadiyne linkage of **192** was undertaken but surprisingly, resisted conversion to the saturated **193**.



193 **192** **194**

Scheme 47. Synthetic attempts at modifying the butadiyne bridge of ***192*** *to produce crystalline derivatives.*

After the failure to produce a crystalline derivative of **192** through modification of the alkyne linkages, it was decided to introduce functionality in the center benzene ring for this purpose. Two 1,4-dibromobenzene derivatives were constructed bearing ester moieties (Scheme 48). Starting from 2-bromobenzoic acid, a second bromide was introduced by electrophilic bromination to produce **196** in 50% yield. The corresponding methyl ester was synthesized by subjecting the acid to MeI and Na_2CO_3 in DMF. The ester, **197**, was obtained as a clear, crystalline solid in 63% yield. Similarly, the *para*-nitrobenzoic ester **198** could be produced by *in situ* formation of the corresponding acid chloride and subsequent exposure to 4-nitrophenol and DMAP to give **198** in 53% yield.

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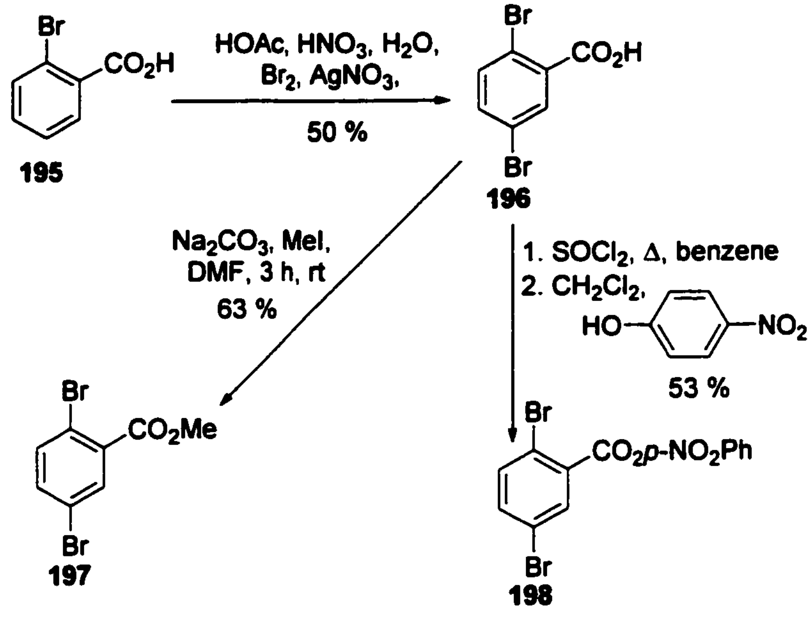
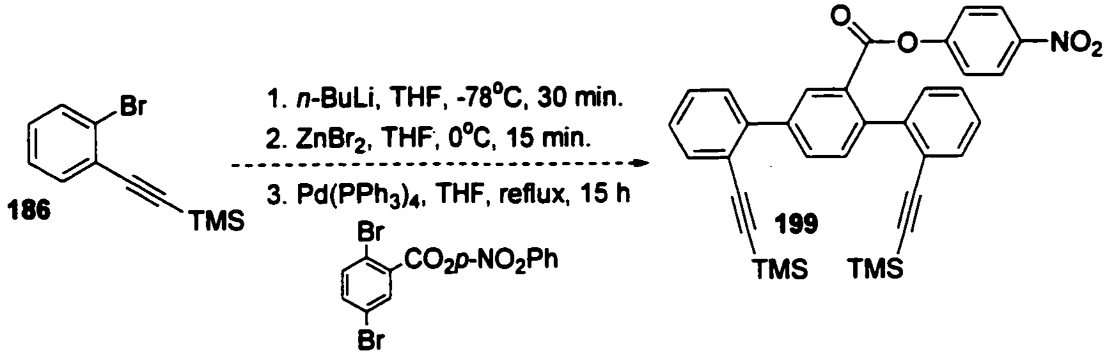
[97] Seyfurth, D.; Kugita, T.; Rheingold, A.L.; Yap, G.P.A. *Organometallics* **1995**, *14*, 5362.



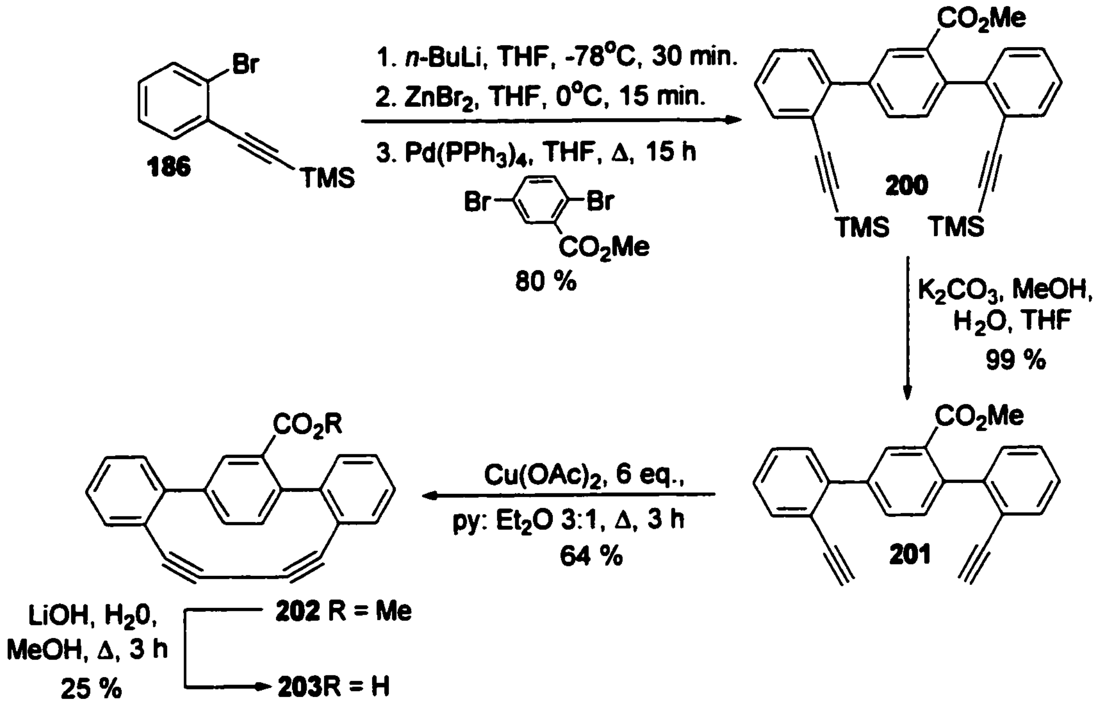
Scheme 48. Chemical modification of the central 1,4-dibromobenzene cyclophane cores.

Dibromide, **198**, did not undergo successful Negishi coupling, perhaps due to the instability of its *p*-nitrophenol ester linkage (Scheme 49).



*Scheme 49. Negishi coupling of **186** to the dibromide, **198** failed.*

The Negishi coupling of **186** with the methyl ester, **197**, produced the precursor, **200** in 80% yield (Scheme 50). Removal of the TMS protecting groups allowed for formation of the terminal acetylenes in quantitative yield. Exposure of **201** to the $Cu(OAc)_2$-mediated dimerization conditions in refluxing 3:1 pyridine:diethyl ether, cyclization occurred to give the monomer product **202** in 64% yield. Although **202** was isolated as a yellow solid, crystallization attempts were not successful. Hydrolysis of the methyl ester was problematic and optimized conditions were developed. Heating was required for hydrolysis, and, long reaction times caused decomposition of the starting material. The acid, **203**, was recrystallized as clear needles by slow evaporation of a dichloromethane solution. (Scheme 50)



*Scheme 50. Synthesis of a crystalline derivative of **203**.*

The X-ray crystal structure revealed the strained nature of the butadiyne bridge (Figure 28). The bonds between C_4-C_{23} and C_7-C_8 are bent ~18° from the plane of the central benzene ring. The triphenyl core is not linear but bowed. The bending in these bonds is most likely a result of the strain apparent in the diacetylene linkages. The acetylene bonds are also strained. The angles between carbon atoms C_{14}-C_{15}-C_{16} and C_{15}-C_{16}-C_{17} are 163.7° and 163.5° respectively.

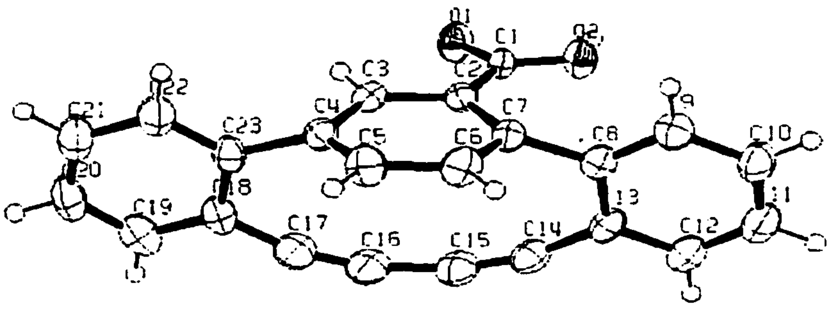


*Figure 28. X-ray crystal structure of **203** reveals the strained butadiyne bridge and the bowed triphenyl core.*

The carboxyl group was introduced to aid crystallization through the formation of a dimer by hydrogen bonding. Examination of the crystal packing structure reveals this is the case (Figure 29). The stereoviews depicted in Figure 29 show that the carboxyl group sits at approximately right angles from the butadiyne bridge. Although within the lattice, the acid group is pointing in the same direction as the butadiyne bridge of an adjacent molecule, it is also hydrogen-bonded to another carboxylic acid.

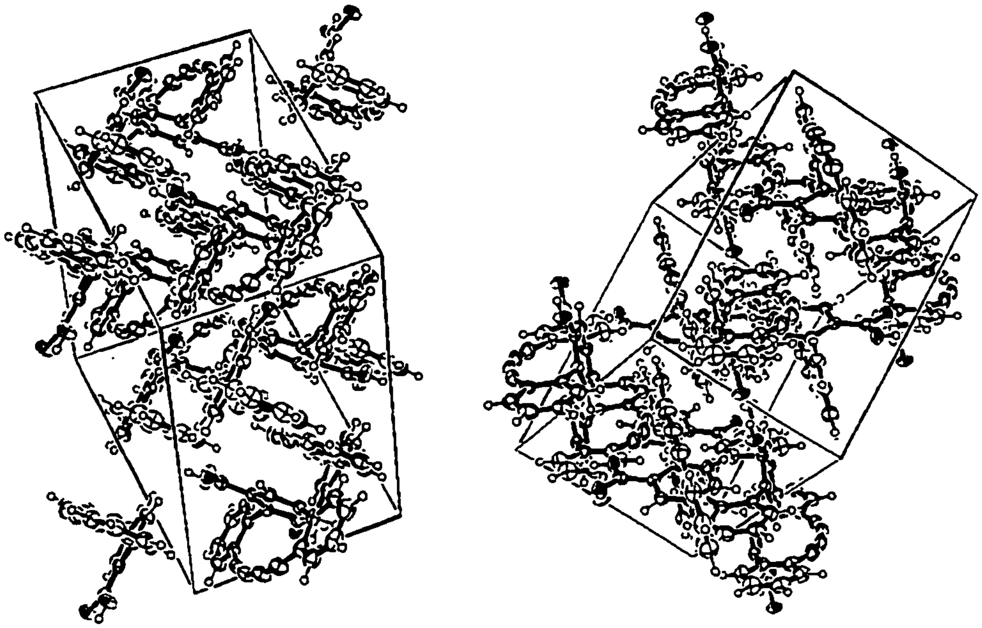
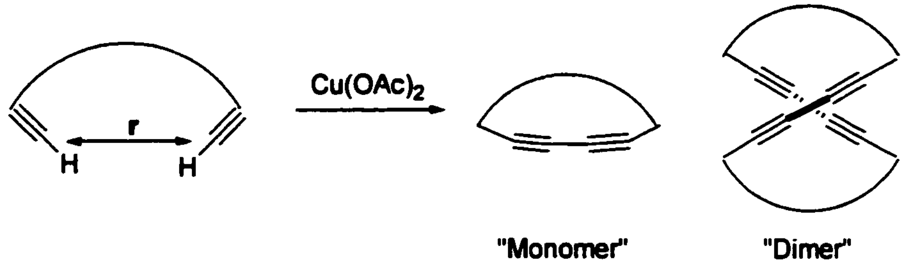


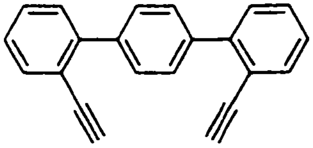
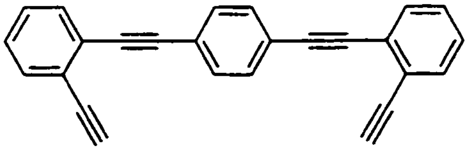
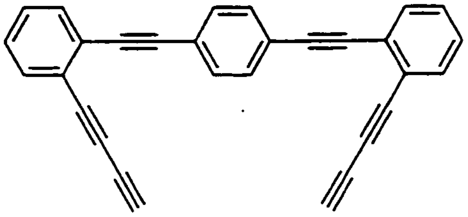
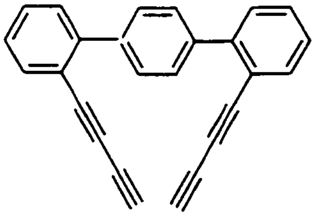
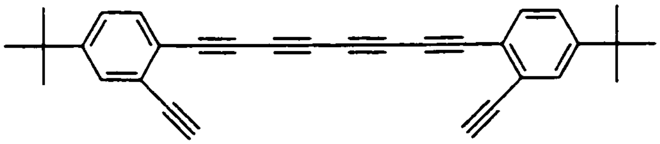
Figure 29. Crystal packing diagram of ***203****.*

3.2 Intra- vs. Intermolecular Cyclization: The Role of Termini Separation.

Examination of the relevant literature concerning the syntheses of large acetylenic cyclophanes suggested that the accepted method of preparation of multibridged macrocycles is through the dimerization of acetylenes. Furthermore, many authors do not report any competing intramolecular coupling products. Consequently, the formation of the "monomer" product, **192** was unexpected. Many of the cyclophanes reported possessed extra acetylene units between the central benzene ring and the outer unsaturated unit. It is possible that these extra acetylene units place the reactive acetylene termini at sufficient distance to inhibit intramolecular coupling. Although not he limiting factor, the distance must be sufficient to render intramolecular coupling improbable since cyclophane **192** suggests that, when possible, intramolecular coupling may predominate to form highly strained products. Table 6 summarizes some calculated separations between two terminal acetylene units in related precursors.

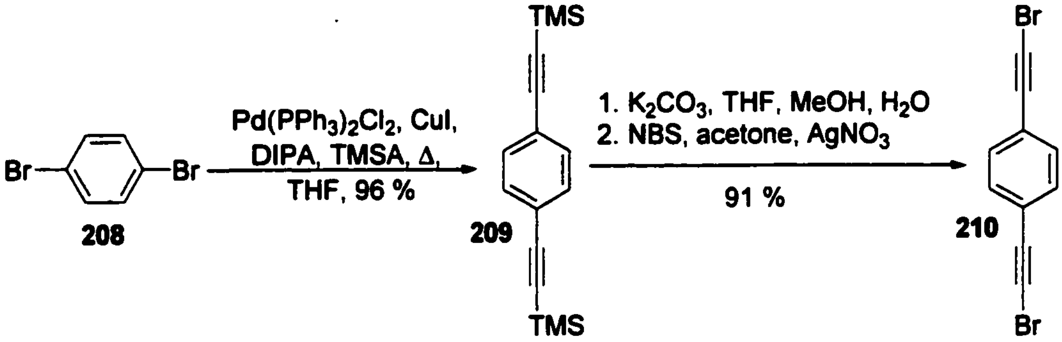
Table 6. Calculated distances between reactive acetylene termini in related [1.4]cyclophanes.



Entry	Substrate	Compound	Distance (Å)
1		**190**	5.03
2		**204**	10.43
3		**205**	4.69
4		**206**	4.58
5		**207**	10.78

The compound **204** represents an extended version of **190** whereby another acetylene spacer was inserted between the phenyl rings (entry 2). The reactive terminal acetylene distance doubled from 5.03 to 10.43 Å. However, the addition of another alkyne unit within the bridging unit would shorten the distance to 4.69 Å (entry 3). At that distance, it is expected that dimerization would cause a product ratio similar to that observed for **190**. Removing the spacers between phenyl rings but keeping the butadiyne linkages would again shorten the closest intramolecular distance to 4.58 Å. Although this distance is shorter than **190**, it is not expected to undergo intramolecular coupling due to the highly strained product that would be formed. Haley and co-workers have demonstrated that dimerization of a *t*-butyl substituted tetrayne, produced solely the dimer product (entry 5).[98] The calculated distance between termini was estimated at 10.78 Å, similar to that calculated for **207** (entries 5 and 2 respectively).

Accordingly, the acetylenic precursor, **204**, a derivative of **190** in which an extra alkyne unit is connected between the benzene rings was synthesized and was expected to give dimer, **213** upon copper mediated oxidation. Two equivalents of TMSA were cross-coupled with 1,4-dibromobenzene to give **209** in 96% yield. The compound was deprotected using potassium carbonate in THF/MeOH, and the terminal diacetylene was reacted in acetone with NBS and a catalytic amount of $AgNO_3$ to give the brominated product, **210** in 91% yield (Scheme 51).



Scheme 51. Synthesis of 1,4-Bis-bromoethynylbenzene.

The organozincate of **185** or **186** could then be generated *via* halogen-metal exchange and transmetalation and then coupled to **210** to give the extended precursor **211** or **212** in 54% or 49%, respectively (Scheme 52). Deprotection of the TIPS protecting group with TBAF or the TMS protecting group with basic potassium carbonate gave the terminal acetylene, **204** in 90% yield. Dimerization of the terminal acetylene by addition of **204** to a solution of $Cu(OAc)_2$ in 3:1 pyridine: diethyl ether produced the dimer, **213** as the only isolatable product in 21% yield.

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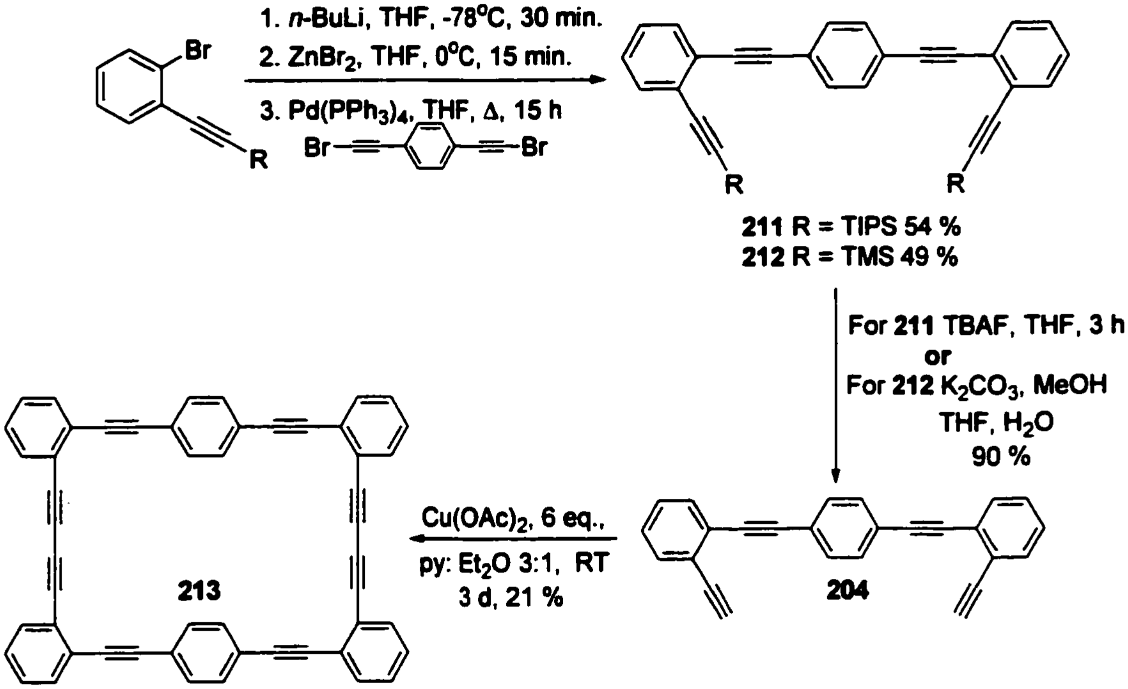
[98] Haley, M.M.; Bell, M.L.; Brand, S.C.; Kimball, D.B.; Pak, J.J.; Wan, W.B. *Tetrahedron Lett.* **1997**, *38*, 7483.



Scheme 52. Synthesis of the extended dimer, 213 by oxidative dimerization of 204.

Crystals of dimer, **213**, were obtained by slow evaporation of a layered mixture of methanol on top of dichloromethane. The dimer began to slowly polymerize over a period of days, and surprisingly, crystals grew adhering to the polymer surface. The structure is depicted in different stereoviews in Figure 30. The structure is similar to that obtained for **191** in that it is also twisted helically with the central benzene rings slightly offset but overlapping one another. The pincer-like shape is lessened compared to **191** and does not co-crystallize with the solvent.

The X-ray crystal-packing diagram revealed that the molecules line up to form columns or rows of molecules in parallel. In this case, the "arms" or corners of the dimer fit into the open space within an adjacent molecule. The packing resulted in a zig-zag layering to the lattice depicted in Figure 31.

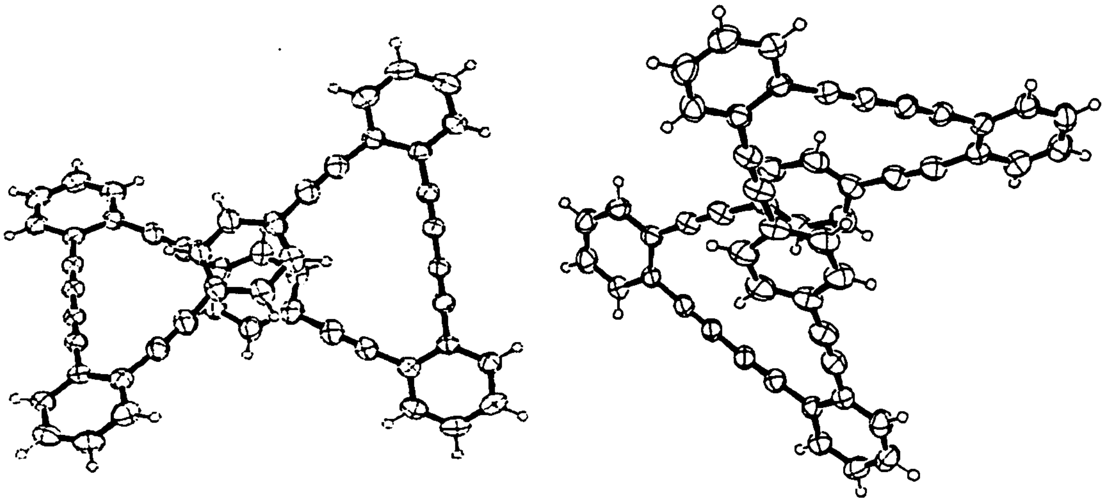


*Figure 30. Two stereoviews of the X-ray crystal structure of the dimer, **213**, from above (left) and from the side (right).*

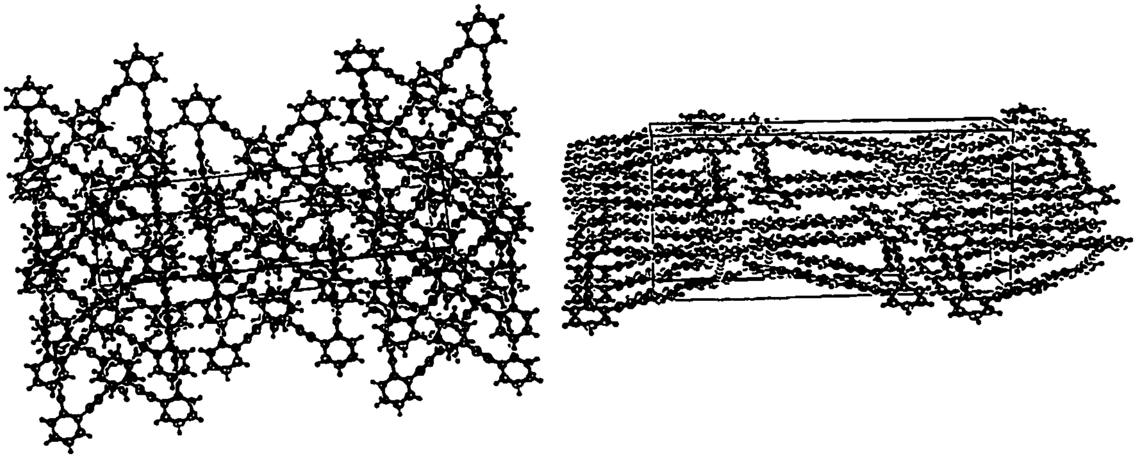
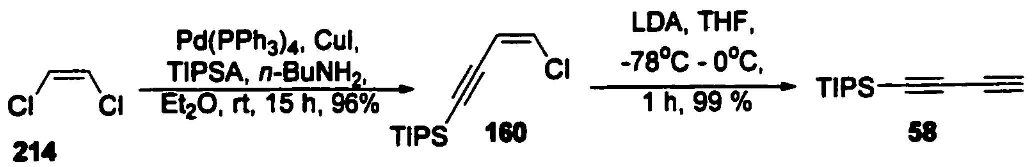


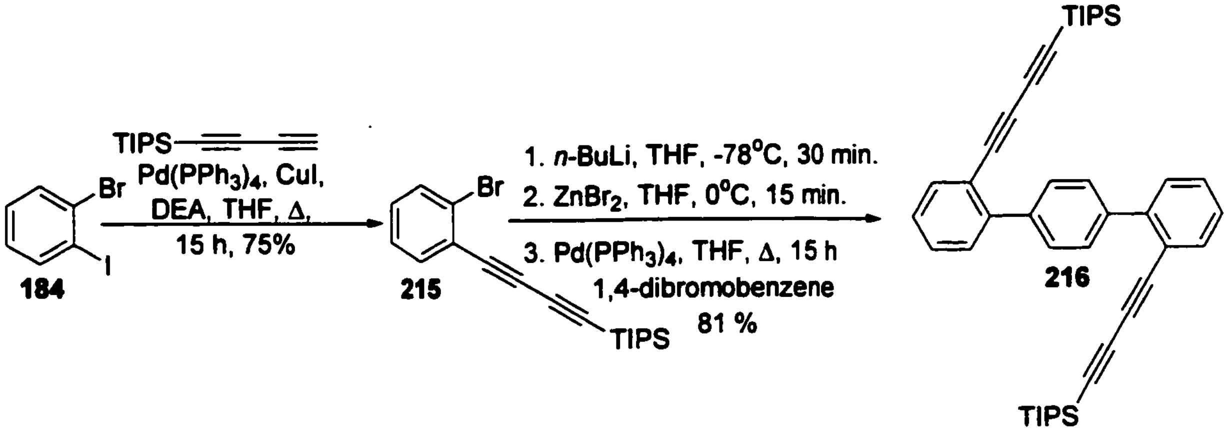
*Figure 31. Two stereoviews of the crystal-packing diagram of **213**.*

The molecule, **206** was also synthesized to test whether the termini separation would dictate the formation of intra- or intermolecular products. The precursor, **206** was expected to give the dimer due to the expected strain in an intramolecular cyclization. The synthesis of 1-(triisopropylsilyl)-1,4-butadiyne is described in Scheme 53. The chloro ene-yne, **160** was synthesized as previously reported by this laboratory. Treating **160** with LDA at room temperature caused no reaction but, when the temperature was lowered to –78 °C and slowly raised to 0 °C, LDA effected elimination of the chloride ion and the lithium diacetylide was quenched with water to give **58** in quantitative yield. The crude compound slowly reddened over a period of minutes and it was best to use **58** immediately after isolation.



Scheme 53. Synthesis of 1-(triisopropylsilyl)-1,4-butadiyne.

Sonogashira coupling of **58** with 1–iodo-2-bromobenzene afforded **215** in 75% (Scheme 54). If compound **215** was stored at –30 °C overnight and the Sonagashira coupling was performed the following day, the yield dropped to less than 30% yield. Generation of the organozincate and palladium catalyzed cross-coupling with 1,4-dibromobenzene gave the precursor **216** in excellent yield (81%). Unfortunately, all attempts to deprotect the compound resulted in decomposition. This is not surprising as terminal phenylbutadiynes are particularly sensitive.[4] For this purpose, a special synthetic protocol would have to be developed for the dimerization of terminal phenyl butadiynes.



*Scheme 54. Construction of **216** by Negishi coupling.*

3.3 Synthesis of Diynes and Tetraynes by an in situ Desilylation/Dimerization Strategy.

The unique electrical, optical, and structural properties of polyynes and acetylenic arrays continue to find potential applications in both biological[99] and material sciences.[12] Consequently, research into the synthesis of well-defined polyynes continues to expand. The electronic properties and spectra of polyynes and polyyne polymers[100] have been

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[99] Faulker, D.J. *Nat. Prod. Rep.* **1995**, *12*, 223 and earlier reviews cited therin.

[100] (a) Shirakawa, H.; Louis, E.J.; MacDiarmid, A.G.; Chiang, C.K.; Heeger, A.J. *J. Chem. Soc., Chem. Comm.* **1977**, 578. (b) Chiang, C.K.; Fincher, C.R. Jr.; Park, Y.W.; Heeger, A.J.; Shirakawa, H.; Louis, E.J.; Gau, S.C.; MacDiarmid, A.G. *Phys. Rev. Lett.* **1977**, *39*, 1098.

studied for their interesting bathochromic shifts.[101] These shifts are proportional to the number of acetylene linkages. [102] α-ω-Diarylpolyynes display efficient charge transfer through their conjugated polyyne bridges in applications as dyes and pigments.[103] The efficiency of the energy and electron transfer processes have been examined as potential molecular wires and chemosensors, particularly in polyyne-bridged porphyrin systems[104] and bis(benzocrown ethers).[105]

Polyyne segments in both natural[106a] and unnatural[8b,c] products exhibit a wide range of biological activity. These include potential as potent anti-inflammatory,[107a] antibiotic[13b] (caryoynencins), antitumour[13c] (panaxydol) and antibacterial[13d] agents (falcarrindiol).

The most common synthetic method for the assembly of polyynes involves bond formation between two acetylenes *via* oxidative coupling. Frequently, the classic protocols developed by Hay,[11] Glaser,[9] and Eglinton[10] are the methods of choice, although newer combinations and modifications have also been developed.[108] Diederich and co-workers have introduced a solution spray flash vacuum pyrolysis of suitable precursors for the formation of even and odd numbered polyynes.[109]

The instability of simple and complex polyynes is a major challenge in their preparation. They are highly sensitive to polymerization and prone to rapid decomposition.[110] Recently, different protocols have been developed for the *in situ* one pot desilylation/dimerization of acetylenes. Mori, Hiyama, and co-workers[111] employed

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[101] Akiyama, S.; Nakasuji, K.; Akashi, K.; Nakagawa, M. *Tetrahedron Lett.* **1968**, *24*, 1121.

[102] Prabhakara, R.; Mathur, S.C.; Dube, D.C.; Tewari, D.P.; Banerjee, M. *Can. J. Chem.* **1997**, *75*, 1041.

[103] Akiyama, S.; Nakashima, K.; Nakatsuji, S.; Nakagawa, M. *Dyes Pigm.* **1990**, *13*, 117.

[104] Maruyama, K.; Kawabata, S. *Bull. Chem. Soc. Jpn.* **1990**, *63*, 170.

[105] Lee, L-H.; Lynch, V.; Lagow, R.J. *J. Chem. Soc., Perkin Trans. 1* **2000**, 2805.

[106] (a) Svirskaya, P.I.; Leznoff, C.C.; Roelofs, W.L. *Synth. Commun.* **1980**, *10*, 391 (b) Rodbotten, S.; Benneche, T.; Undhiem, K. *Acta Chem. Scand.* **1997**, *51*, 873. (c) Hopf, H.; Krause, N. *Tetrahedron Lett.* **1985**, *26*, 3323.

[107] (a) Zeni, G.; Panatieri, R.B.; Lissner, E.; Menezes, P.H.; Braga, A.L.; Stefani, H.A. *Org. Lett.* **2001**, *3*, 819. (b) Yamaguchi, M.; Park, H.-J.; Hirama, M.; Torisu, K.; Nakamura, T.; Minami, H.; Nishihara H.; Hriraoka, T. *Bull. Chem. Soc. Jpn.* **1994**, *67*, 1717. (c) Lu, W.; Haji, G.Z.; Cai, J. *Tetrahedron Lett.* **1998**, *39*, 9521. (d) Zheng, G.; Lu, W.; Cai, J. *J. Nat. Prod.* **1999**, *62*, 626.

[108] (a) Siemsen, P.; Livingston, R. C.; Diederich, F. *Angew. Chem. Int. Ed.* **2000**, *39*, 2632. For additional palladium based alkyne coupling methods see (b) Liu, Q.; Burton, D.J. *Tetrahedron Lett.* **1997**, *38*, 4371. (c) Godt, A. *J. Org. Chem.* **1997**, *62*, 7471.

[109] Rubin, Y.; Lin, S.S.; Knobler, C.B.; Anthony, J.; Boldi, A.M.; Diederich, F. *J. Am. Chem. Soc.* **1991**, *113*, 6943.

[110] Patel, G.N.; Chance, R.R.; Turi, E.A.; Khanna, Y.P. *J. Am. Chem. Soc.* **1978**, *100*, 6644.

[111] Ikegashira, K.; Nishihara, Y.; Hirabayashi, K.; Mori, A.; Hiyama, T. *Chem. Commun.* **1997**, 1039.

CuCl in DMF under air or oxygen with trimethylsilylacetylene in the absence of fluoride ion to afford high yields (80–100%) of the homocoupled product. This procedure has also been applied to the synthesis of unsymmetrically substituted diacetylenes when alkynyltrimethylsilanes are mixed with 1-chloroalkynes.[112] Haley *et al.* reported a different *in situ* one pot desilylation/dimerization strategy for the synthesis of tetrayne linked dehydrobenzoannulenes. This method combines standard Eglinton coupling conditions with an excess of potassium carbonate to effect the protodesilylation of trimethylsilyl (TMS) protected acetylenes in good yields.[113]

Our synthesis of acetylenic precursors involved the use of triisopropylsilyl (TIPS) protected phenylbutadiyne units. Deprotection and isolation of the terminal acetylenes was not possible as the products rapidly decomposed before oxidative coupling could occur. In order to extend the studies of acetylenic cyclophanes to tetrayne-bridged systems, an alternative desilylation/dimerization protocol was developed using a fluoride source to effect desilylation of TIPS protected acetylenes.[114]

Initially, the dimerization of model compounds **217** and **218** (Table 7) was investigated. Compound **217** was stirred with a fluoride source (TBAF)[115] and $Cu(OAc)_2$ in pyridine/ether (3:1) for 4 hours to give a 68% yield of the diyne product. Doubling the concentration of **217** did not increase the yield (Entry 2). It appeared that the deprotected phenylbutadiyne was polymerizing rapidly, and thus competing with the desired dimerization. Consequently, controlled addition of substrate **217** *via* syringe pump was attempted (Entry 3). Unfortunately, under identical concentrations, yields comparable to those without controlled addition were obtained. An increase in the number of equivalents of fluoride ion also had no effect (Entry 4). Exposure of **217** to CuF_2 as a combined source of fluoride ion for desilylation and copper ion for oxidative coupling also proved futile. Copper (II) fluoride is insoluble in common organic solvents and under heterogeneous reaction conditions only the starting material was recovered.

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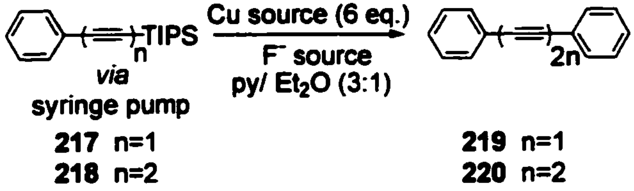
[112] Nishihara, Y.; Ikegashira, K.; Mori, A.; Hiyama, T. *Tetrahedron Lett.* **1998**, *39*, 4075.

[113] Haley, M.M.; Bell, M.L.; Brand, S.C.; Kimball, D.B.; Pak, J.J.; Wan, W.B. *Tetrahedron Lett.* **1997**, *38*, 7483.

[114] Heuft, M.A.; Collins, S.K.; Yap, G.P.A.; Fallis, A.G. *Org. Lett.* **2001**, *3*, 2883.

[115] (a) Oda, H.; Sato, M.; Morizawa, Y.; Oshima, K.; Nozaki, H. *Tetrahedron* **1985**, *41*, 3257. (b) Johnson, T.R.; Walton, D.R.M. *Tetrahedron* **1972**, *28*, 5221.

Table 7. Homocoupling of ***217*** *and* ***218*** via *an in-situ Desilylation/dimerization strategy.*



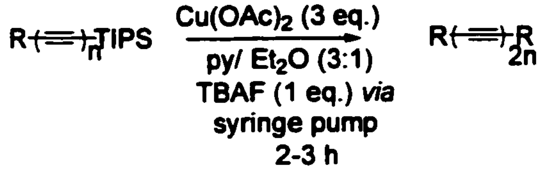
217 n=1
218 n=2

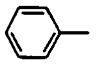
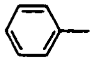
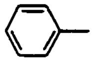
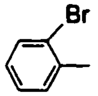
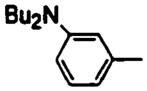
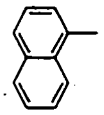
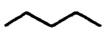
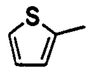
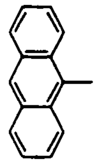
219 n=1
220 n=2

Entry	[mM]	Copper source	F^- source	Time (h)	Yield (%)
1	**217**, 2.0	$Cu(OAc)_2$	TBAF 1 eq.	4	**219**, 68[a]
2	**217**, 4.0	$Cu(OAc)_2$	TBAF 1 eq.	4	**219**, 45[a]
3	**217**, 4.0	$Cu(OAc)_2$	TBAF 1 eq.	3	**219**, 42
4	**217**, 4.0	$Cu(OAc)_2$	TBAF 2 eq.	2	**219**, 45
5	**217**, 4.0	CuF_2	-	2	NR[b]
6	**218**, 2.0	$Cu(OAc)_2$	TBAF 1 eq.	2	**220**, 15-40
7	**218**, 2.0	$Cu(OAc)_2$	CsF 1 eq.	3	**220**, 6
8	**218, 2.0**	**$Cu(OAc)_2$**	**TBAF** **1 eq.**	**3**	**220, 100**[c]
9	**218**, 2.0	CuCl, TMEDA	TBAF 1 eq.	2	**220**, < 5[d]

[a] substrate not added by syringe pump
[b] NR = no reaction
[c] TBAF added via syringe pump instead of substrate
[d] benzene used as solvent

Table 8. Homocoupling of various butadiynes via an in situ Desilylation/dimerization strategy.



Entry	R	n	[Substrate] mM	Yield (%)
1		2	**218**, 1.7	**220**, 73
			218, 3.3	**220**, 100
2		1	**217**, 3.3	**219**, 100
3		2	**221**[a], 3.3	**220**, 93
4		2	**215**, 3.3	**222**, 98
5		2	**223**, 3.3	**224**, 82
6		2	**225**, 3.3	**226**, 96
7		2	**227**, 3.3	**228**, 92
8		2	**229**, 3.3	**230**, 82
9		3	**231**, 3.3	-

[a] TMS group in place of a TIPS group.

The generality of this method was established by investigation of the dimerization of compounds listed in Table 8. Model compound **218** at various concentrations was exposed to TBAF. A concentration of 1.7 mM afforded the isolated tetrayne product,

220, in 73-77% yield. However, doubling the concentration of **218** gave quantitative yields. The use of a TMS silyl protected diyne (**221**, entry 3) also gave, as expected, the diaryl tetrayne in 93% yield. Substituent effects were also investigated. Compound **215**, which has an electron withdrawing *o*-bromo phenyl, was dimerized in 98% yield. X-ray crystallographic analysis established the structure of the tetrayne (**222**). This material displayed crystal packing similar to the system reported by Tykwinski and co-workers for cross-conjugated polyenynes (Figure 32).[116] The electron rich *m*-N,N-dibutylaminophenyl diyne **223** formed the corresponding tetrayne in 82%. Desilylation/dimerization of **225** afforded the known tetrayne 1,8-(1-naphthyl)-octa-1,3,5,7-tetrayne, **226**, 96%, previously investigated for its interesting spectral properties.[102] Alkyl substituents were also compatible with this synthetic protocol as **227** dimerized in 98%. Heteroaromatic substituted polyynes may also be easily prepared by this procedure. For example, **229** produced the thiophene-tetrayne (**230**) in 82% yield.[103]

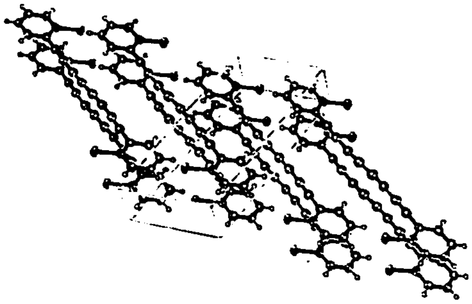


*Figure 32. View of the unit cell crystal packing for the 1,8-di-(2-bromophenyl)-1,3,5,7-octatetrayne, **222**.*

An attempt to form a hexayne product *via* this procedure failed. The anthracenyl triyne **231** was constructed in a manner parallel to that described by Tykwinski and co-workers (Scheme 55).[117] Sonogashira coupling of TMSA with 9-bromoanthracene followed by desilylation was accomplished in quantitative yield. The corresponding

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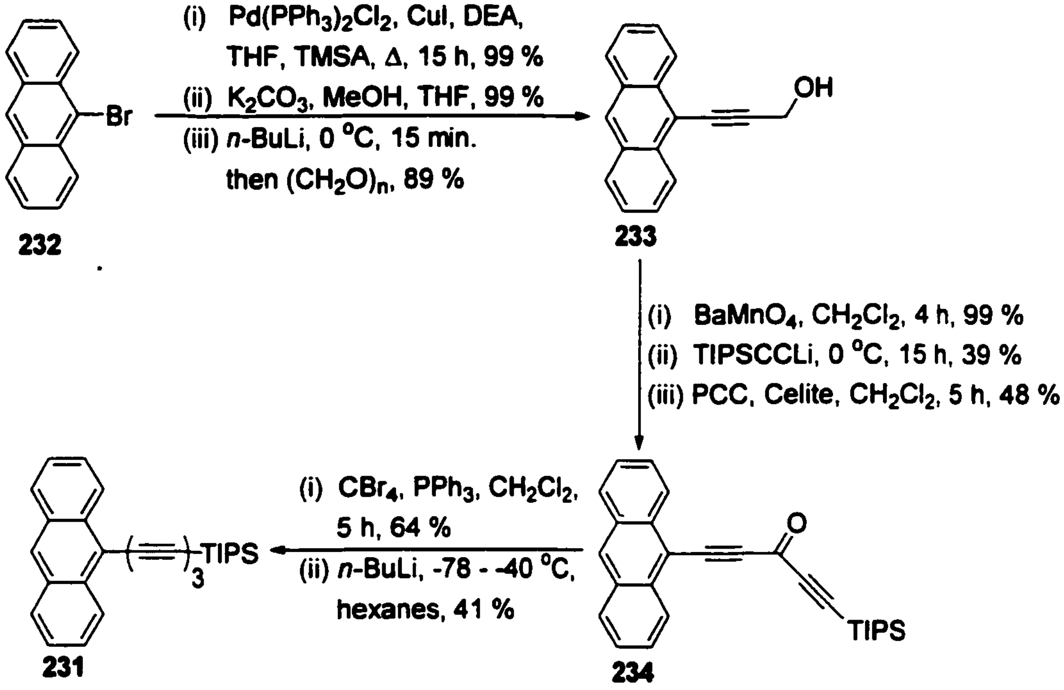
[116] Zhao, Y.; McDonald, R.; Tykwinski, R.R. *Chem. Commun.* **2000**, 77.

[117] Compound **231** was prepared from a Fritsch-Buttenberg-Wiechell (FBW) rearrangement (recent applications: Eisler, S.; Tykwinski, R.R. *J. Am. Chem. Soc.* **2000**, *122*, 10736).

terminal acetylene was deprotonated with *n*-BuLi and the resulting acetylide was quenched with *p*-formaldehyde to give the propargyl alcohol, **233** in 89% yield. Oxidation of **233** with freshly prepared $BaMnO_4$ in dichloromethane gave the corresponding aldehyde in 99% yield. The lithium salt of TIPSA was generated in THF by adding one equivalent of *n*-BuLi to the triisopropylsilylacetylene at 0 °C followed by the addition of the aldehyde. After stirring at room temperature for 15 hours, the resulting alcohol was isolated and oxidized to the ketone, **234** with PCC in dichloromethane in 48% yield. After the ketone was converted to the corresponding dibromide *via* a Corey-Fuchs reaction, the dibromide was cooled to –78 °C, and treated with *n*-BuLi and the solution slowly warmed to –40 °C. A Fritsch-Buttenberg-Wiechell (FBW) rearrangement produced the triyne, **231** in 41% yield. The triyne, **231** (Entry 9) gave two red solid products upon treatment with the $TBAF/Cu(OAc)_2$ system. Both compounds were sparingly soluble in organic solvents. At present, these cannot be properly identified or characterized.

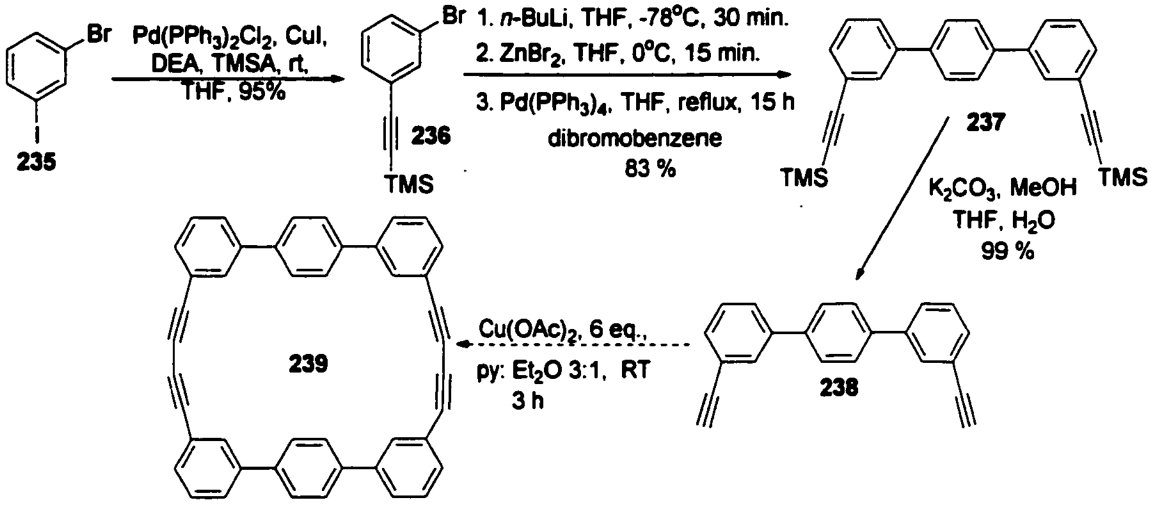


Scheme 55. Synthesis of the triyne, 231.

The optimized desilylation/dimerization conditions were then applied to the dimerization of **216**. Despite the fact that other tetrayne linked acetylenic cyclophanes have been synthesized using the developed protocol,[118] no formation of dimer was observed at substrate concentrations of 3.3 and 1.65 mM.

3.4 Synthesis of Acetylenic Cyclophanes with meta-Phenyl-Yne Linkages.

With the success in synthesizing acetylenic cyclophanes bearing *ortho*-phenyl-yne linkages, the design of a second family bearing *meta*-phenyl-yne bridges was investigated. Sonogashira coupling of TMSA with 3-bromo-1-iodobenzene generated the *meta*-phenyl-yne precursor, **236** in 95% yield. Generating the organozincate of **236** followed by palladium-catalyzed cross-coupling to 1,4-dibromobenzene gave the TMS protected product, **237** in 83% yield (Scheme 56). The TMS protecting groups were removed under basic conditions with potassium carbonate in MeOH and THF to give **238** in quantitative yield. The diacetylene, **238**, was then added by syringe pump to achieve pseudo high dilution conditions to minimize polymerization of the starting material. After addition to a solution of $Cu(OAc)_2$ in 3:1 pyridine: diethyl ether no soluble organic products could be isolated.



Scheme 56. Synthetic progress towards the dimer, 239.

The inability of **238** to undergo successful dimerization is not surprising when compared to the work of other groups using *meta*-phenyl linkages. The meta-phenyl-yne structure is an extremely useful monomer in the design of non-biological oligomers.[119] However, their usage in cyclophane chemistry has remained remote and they are often installed by bromination/dehydrobromination of alkenes already present within a

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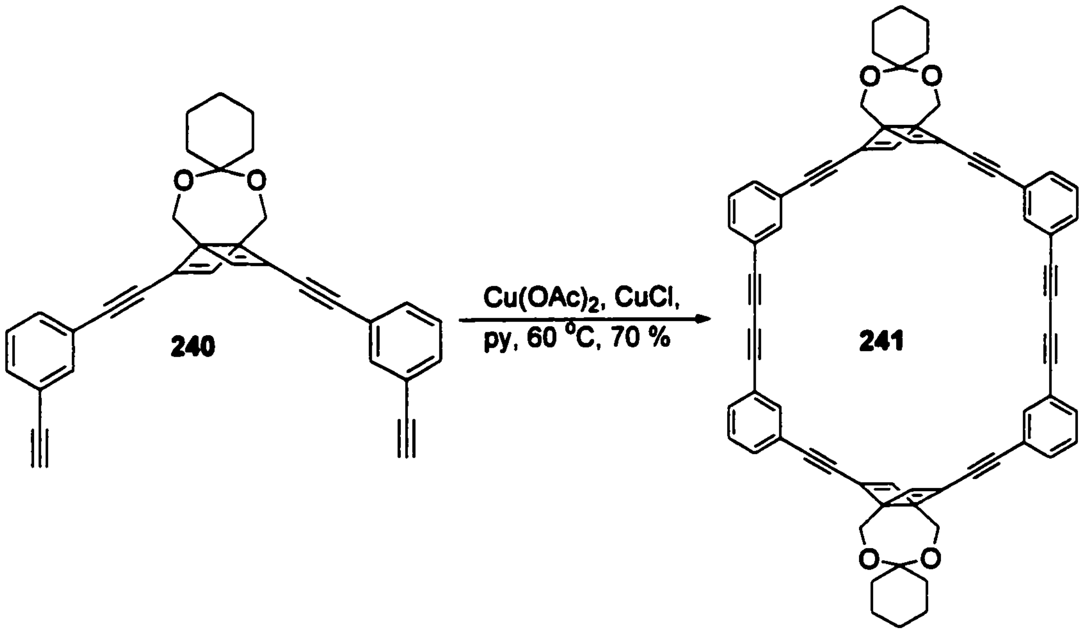
[118] Heuft, M.A.; Collins, S.K.; Yap, G.P.A.; Fallis, A.G. *Org. Lett.* **2001**, *3*, 2883.
[119] Prince, R.B.; Okada, T.; Moore, J.S. *Angew. Chem. Int. Ed. Engl.* **1999**, *38*, 233.

macrocycle.[120] A dimerization of terminal acetylenes strategy has been applied successfully when the triple bond was connected to the phenyl moiety by a heteroatom, permitting an added degree of flexibility.[121] Tsuji and co-workers have dimerized, **240**, and attributed its success to the fact that the central dewar benzene moiety allows for the terminal acetylenes to be orientated in an appropriate geometry for dimerization.[122]



*Scheme 57. Tsuji's construction of the acetylenic cyclophane, **241**.*

In summary, routes to *para*-cyclophanes have been developed, optimized and have resulted in the synthesis of a family of related acetylenic macrocyles. Factors governing the cyclization of suitable precursors to the dimers were investigated and an improved understanding of the molecular packing and three-dimensional structure of the cyclophanes has been achieved.

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[120] Kawase, T.; Ueda, N.; Darabi, H.R.; Oda, M. *Angew. Chem. Int. Ed. Engl.* **1996**, *35*, 1556.

[121] Tashiro, K.; Aida, T.; Zheng, J.-Y.; Kinhara, K.; Saigo, K.; Sakamoto, S.; Yamaguchi, K. *J. Am. Chem. Soc.* **1999**, *121*, 9477.

[122] Ohkita, M.; Ando, K.; Suzuki, Tsuji, T. *J. Org. Chem.* **2000**, *65*, 4385.

CHAPTER 4: SYNTHESIS OF NOVEL ACETYLENIC CYCLOPHANES WITH HELICAL CHIRALITY: POTENTIAL NEW STRUCTURES FOR LIQUID CRYSTALS

4.1 Carbon Networks as Novel Liquid Crystal and Other Optically Active Materials

Although liquid crystals (LCs) have been known for a century or more, it is only in the past twenty years that their unusual properties and colour changes have found applications. Liquid crystals have found widespread application in the manufacturing of controlled display devices such as wristwatches, calculators, television and computer screens. These materials order themselves to a degree to produce a phase neither liquid nor solid. Magnetic and electrical fields or even temperature can alter the nature of the arrangement of the liquid crystal molecules. Liquid crystals have proven to be a great tool in studying chirality. The helical packing of chiral materials in the liquid crystalline state affords special properties, including the ability to rotate polarized light.[123]

The design of liquid crystals, dopants, and optically active materials using chiral aromatic cores has attracted increased interest.[124] The best ferroelectric liquid crystals have an aromatic core adjacent to a chiral center. Enantiomerically pure materials containing biphenyl cores have been investigated for use as liquid crystals as well as dopants.[125] Lemieux and co-workers have developed powerful atropisomeric dopants based on substituted enantiomerically pure biphenyls and induced a ferroelectric S_c* liquid phase when doped into smectic C (S_c) liquid crystals.[126]

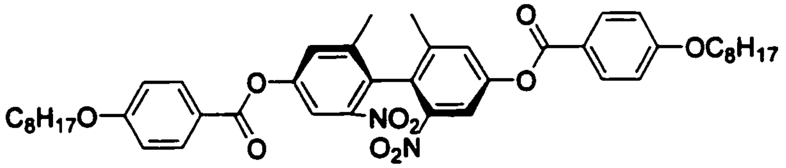

Figure 33. An atropisomeric dopant for the formation of chiral smectic C liquid crystalline phases.

Until recently, there were no examples of liquid crystals in which the aromatic core also provided chirality. However, Katz *et al.* have constructed racemic helicenes

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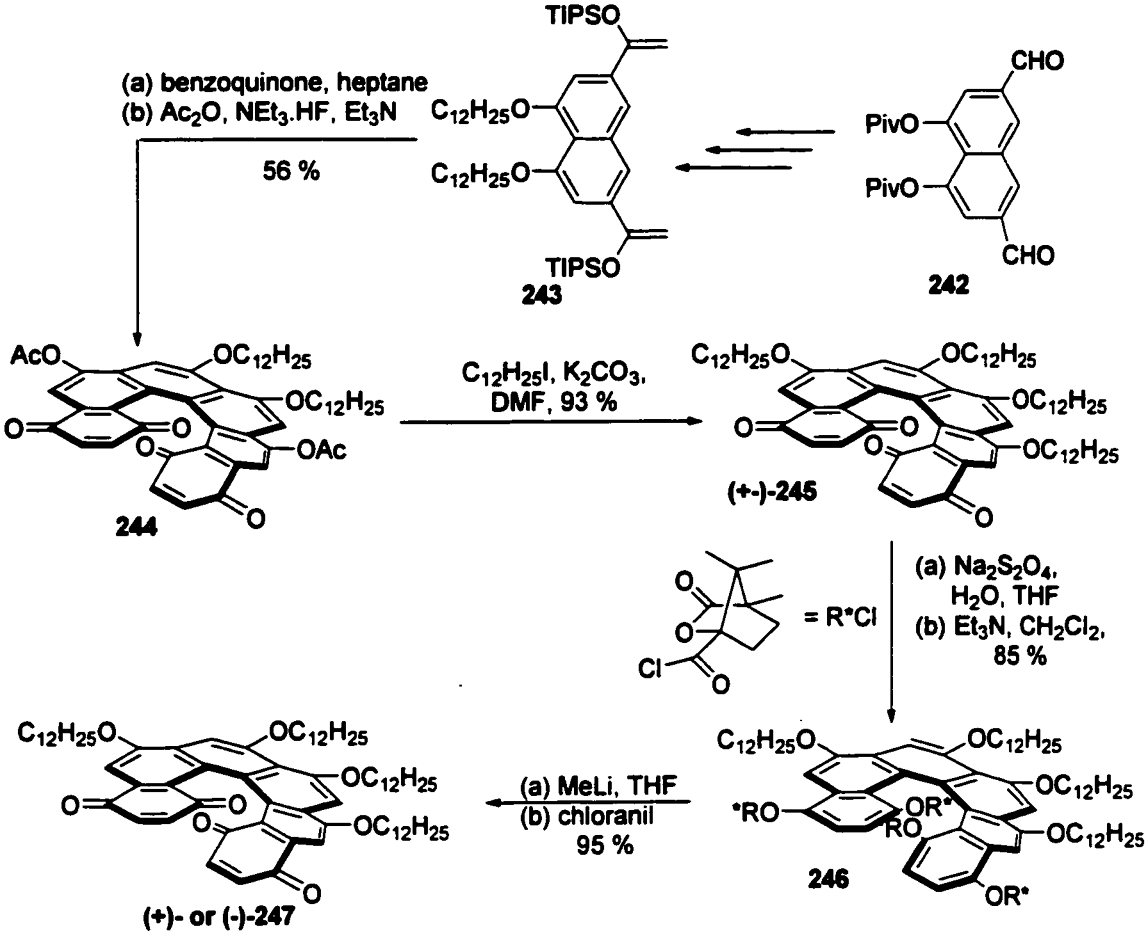
[123] Solladie, G.; Zimmermann, R.G. *Angew. Chem. Int. Ed. Engl.* **1984**, *23*, 348.

[124] Katz, T. J. *Angew. Chem. Int. Ed.* **2000**, *39*, 1921.

[125] Solladie, G.; Hugele, P.; Bartsch, R.; Skoulios, A. *Angew. Chem. Int. Ed. Engl.* **1996**, *35*, 1533.

[126] (a) Vizitiu, D.; Lazar, C.; Halden, B.J.; Lemieux, R.P. *J. Am. Chem. Soc.* **1999**, *121*, 8229. (b) Vizitiu, D.; Halden, B.J.; Lemieux, R.P. *Chem. Commun.* **1997**, 1123.

containing long alkyl chains which form a liquid crystalline phase and assemble in solution to form helical columns.[127] Subsequently, they prepared the related chiral derivatives **247** to generate the first liquid crystalline columnar mesophase molecules with a nonracemic helical aromatic core (Scheme 58).[128]



Scheme 58. Nonracemic helicenes form a liquid crystalline phase and assemble in solution to form helical columns

The value in the synthetic pursuit of π-conjugated and cross conjugated macromolecules with well defined architectures is that it allows one to impart specific properties onto materials *via* their structure (i.e. Structure-properties relationships). Marsella *et al.* have synthesized helical scaffolds based on the thiophene moiety to create long helical like cyclophanes (Scheme 59). Their palladium based coupling strategy was

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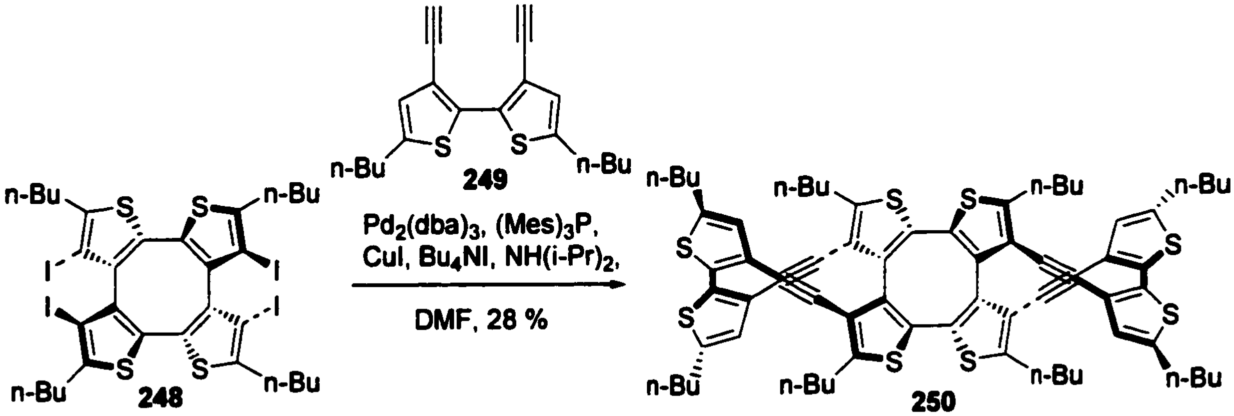
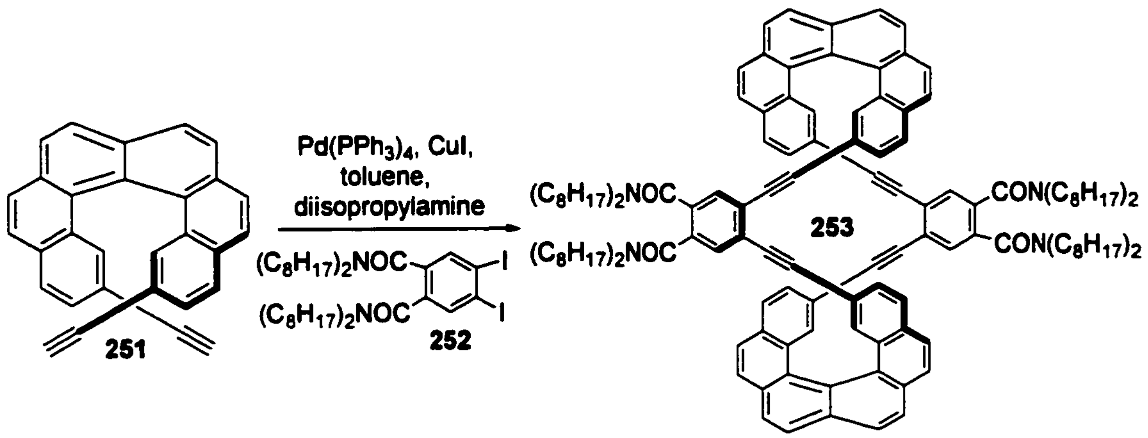
[127] Nuckolls, C.; Katz, T.J.; Katz, G.; Collings, P.J.; Castellanos, L. *J. Am. Chem. Soc.* **1999**, *121*, 79.
[128] Nuckolls, C.; Katz, T.J. *J. Am. Chem. Soc.* **1998**, *120*, 9541.

impressive, as the final step consisted of a tandem Pd/Cu catalyzed cross-coupling to create four new bonds. The four couplings, two of which are intramolecular, occur to give **250** in an overall 28% yield, an average of 73% per coupling. The X-ray structure of the molecule revealed an octa-aryl double helical oligomeric scaffold and a supramolecular double helix along the *c*-axis. This novel aromatic helical core is being investigated as molecular electromagnets and for its novel optical properties.[129]



Scheme 59. Conjugated ladder-type macromolecule exhibiting a double helical motif, capable of creating organic materials with novel optical and eletromagnetic properties.



Scheme 60. Optical properties of nonracemic helicene-cyclophanes differ when compared to binathyls, helicene contaning polymers and polyarylenes.

A number of D_2-symmetric cyclophanes,[130] if constructed to contain a helical twist within their structure, are thought to be novel structures for liquid crystalline

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[129] Marsella, M.J.; Kim, I.T.; Tham, F. *J. Am. Chem. Soc.* **2000**, *122*, 974.

[130] Malaba, D.; Djebli, A.; Chen, L.; Zarate, E.A.; Tessier, C.A.; Youngs, W. J. *Organometallics* **1993**, *12*, 1266.

materials.[131] Helicene containing polymers and cyclophanes have been assembled in order to create a helical twist or geometry within the structures.[132] Fox *et al.* constructed cyclophanes bearing chiral helicenes linked by diethynylbenzene linkages (Scheme 60). The UV and CD spectra of these molecules do not exhibit any noticeable shift to the red as observed for the monomeric analogues. It is postulated that the two helicene rings are twisted with respect to each other and negate any effect.

Disc-like planar carbon networks composed of a rigid core and flexible outward side chains can form columnar mesophases. Macrocyclic compounds such as **254** and **255**, are of special interest as they may form channels in the liquid crystalline phase and may combine to form host/guest complexes.[133] The optical and thermal properties of these compounds are especially sensitive to the position and length of the side chains on the rigid aromatic core. The solid state configuration of **254** reveals that each disc is filled with its own alkyl chains. However, in derivative **255**, the absence of any inward chains creates different solid and solution configuration and thus different LC behavior. In the solution state, the alkyl chains are thought to fold over each side of the cavity of the disc. In contrast, the solid state analysis revealed that the cavity of each disc is filled with the alkyl chains of neighbouring molecules.

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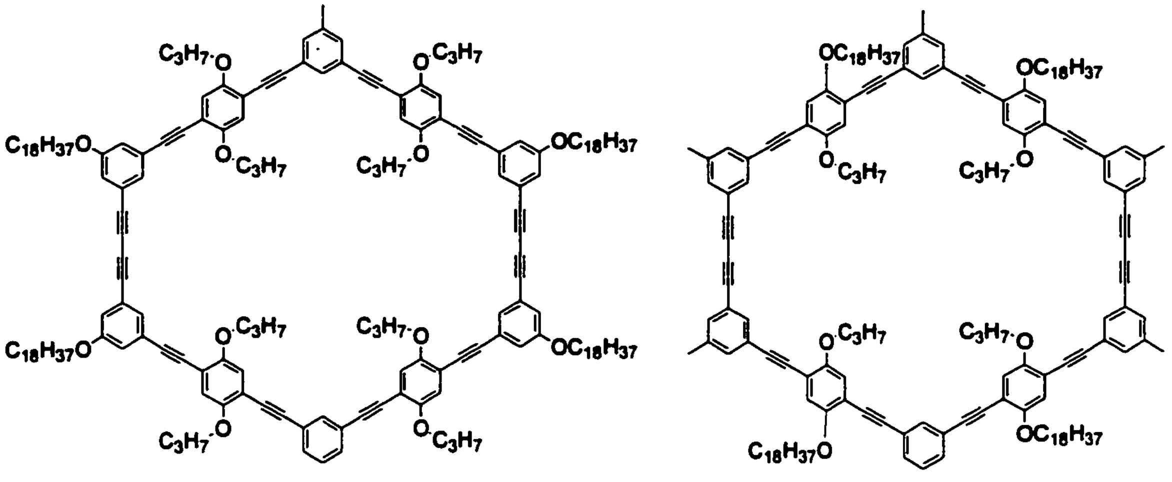
[131] (a) Mizutani, T.; Yagi, S.; Morinaga, Y.; Nomura, T.; Takagishi, T.; Kitagawa, S.; Ogoshi, H. *J. Am. Chem. Soc.* **1999**, *121*, 754. (b) Yang, K.; Campbell, B,; Birch, G.; Williams, V. E.; Lemieux, R. P. *J. Am. Chem. Soc.* **1996**, *118*, 9557. (c) Chin, E.; Goodby, J.W. *J. Am. Chem. Soc.* **1986**, *108*, 4736. (d) Goodby, J. W.; Chin, E., Leslie, T. M.; Geary, J. M.; Patel, J. S. *J. Am. Chem. Soc.* **1986**, *108*, 4729.

[132] Fox, J.M.; Lin, D.; Itagaki, Y.; Fujita, T. *J. Org. Chem.* **1998**, *63*, 2031.

[133] (a) Solladie, G.; Hugele, P.; Bartsch, R.; Skoulios, A. *Angew. Chem. Int. Ed. Engl.* **1996**, *35*, 1533. (b) Hoger, S.; Enkelman, V.; Bonrad, K.; Tschierske, C. *Angew. Chem. Int. Ed. Engl.* **2000**, *39*, 2268.



254 **255**

Figure 34. Disclike molecules composed of a rigid core can form discotic columnar thermotropic mesophases.

4.2 Retrosynthetic Analysis and Design of Helical Aromatic Core For Liquid Crystals.

The design of liquid crystals, dopants and optically active materials using chiral aromatic cores has attracted increased interest.[123] The unsaturated linkages in phenyl acetylene [1.4]paracyclophanes result in a twisted conformation that imparts helical chirality. This property, which is shared with other *D*2-symmetric cyclophanes,[134] should allow these molecules to be employed as novel liquid crystalline materials.[135] It envisioned that modification of our acetylenic cyclophanes should also afford compounds that would exhibit interesting optical properties, and in addition, they might serve as liquid crystal dopants to induce the type of polarization observed for smectic liquid crystals (Figure 35).

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[134] Malaba, D.; Djebli, A.; Chen, L.; Zarate, E. A.; Tessier, C. A.; Youngs, W. J. *Organometallics* **1993**, *12*, 1266.

[135] (a) Mizutani, T.; Yagi, S.; Morinaga, Y.; Nomura, T.; Takagishi, T.; Kitagawa, S.; Ogoshi, H. *J. Am. Chem. Soc.* **1999**, *121*, 754. (b) Yang, K.; Campbell, B,; Birch, G.; Williams, V. E.; Lemieux, R. P. *J. Am. Chem. Soc.* **1996**, *118*, 9557. (c) Chin, E.; Goodby, J.W. *J. Am. Chem. Soc.* **1986**, *108*, 4736. (d) Goodby, J. W.; Chin, E., Leslie, T. M.; Geary, J. M.; Patel, J. S. *J. Am. Chem. Soc.* **1986**, *108*, 4729.

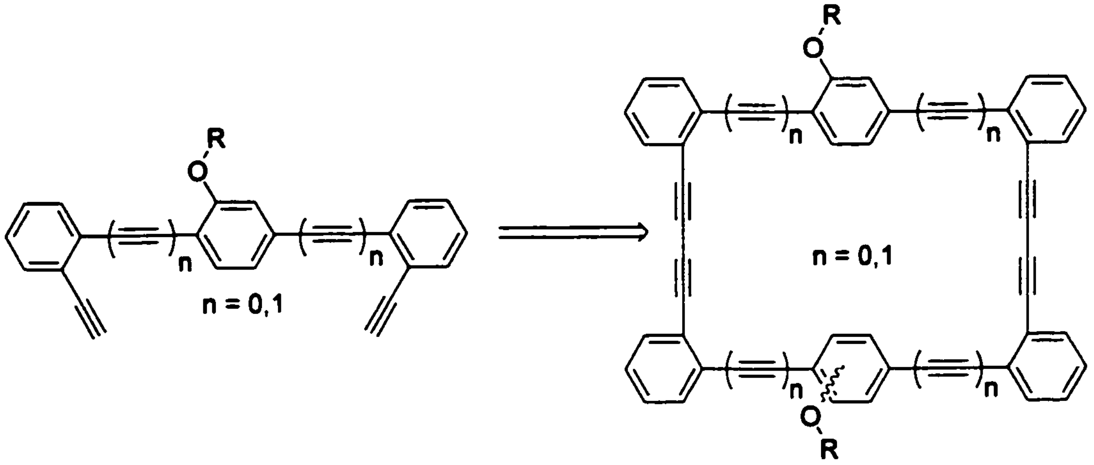


Figure 35. Modification of the aromatic core of phenyl-acetylene produces potential liquid crystals.

The parent ring systems represented by the cyclic structures **191** and **213** appeared to posses the required geometric constraints to provide the desired rigid conformation (Figure 36). The preparations of **191** and **213** have been previously described and analysis of the X-ray crystal structures revealed that the hydrocarbon cores adopt twisted helical geometries. The degree of the twist varies with the overall size of the aromatic core and the number of acetylene units present in the linkages. In both cases, the two central benzene rings are aligned in a slightly offset but overlapping, superimposed manner.

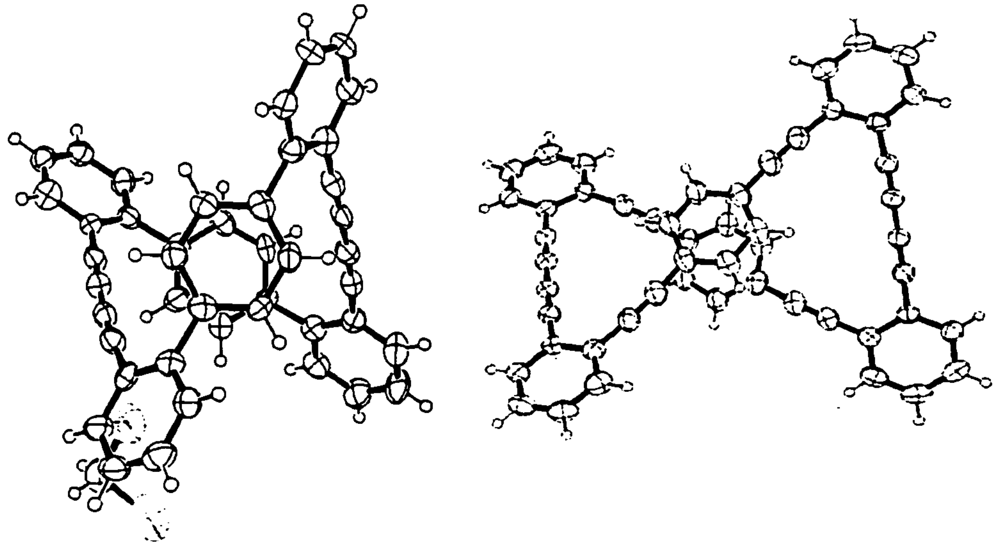
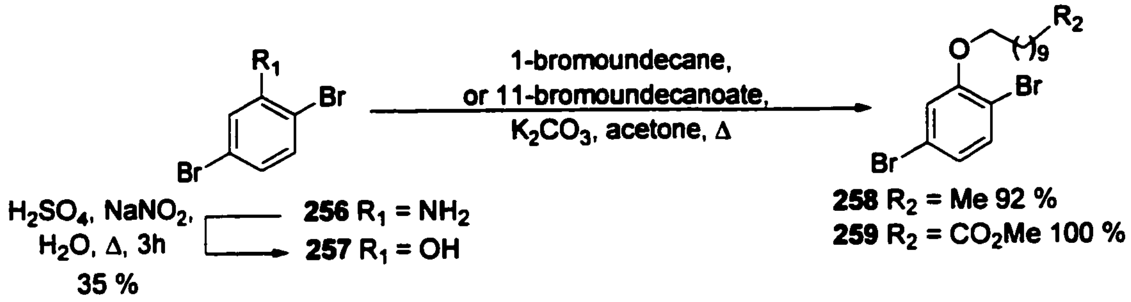


*Figure 36. X-ray crystal structures of **191** and **213** reveal a helical conformation and the potential to use such aromatic cores for potential new liquid crystals.*

Our strategy for the potential liquid crystalline cyclophanes was based upon a synthetic route in which a C_{11}- alkyl chain could be attached to the central benzene ring by an ether linkage. The first series examined were cyclophanes related to **191** (n = 0) in which the acetylene spacers are not present.



Scheme 61. Synthesis of the aromatic dibromide core bearing alkyl chains connected by phenolic ether linkages.

These syntheses commenced with the conversion of 2,5-dibromoaniline to the corresponding phenol *via* a diazonium salt intermediate (Scheme 61). Attachment of the appropriate side chain was accomplished in greater than 90% yield by refluxing the alkyl bromide with the phenol in acetone in the presence of K_2CO_3 (Scheme 61). Compounds **258** and **259** were employed as starting materials for the synthesis of both the shortened (n = 0) and extended (n = 1) cyclophanes.

Construction of the phenyl acetylene precursors was achieved using ZnBr-mediated palladium cross coupling methodology described previously (Scheme 62).[3,136] Generation of the organozincate of (2-bromophenylethynyl)trimethylsilane followed by addition of **258** and $Pd(PPh_3)_4$ afforded the silyl protected derivative **260** in 78% yield. Deprotection of the trimethylsilyl groups gave the diacetylene **261** in quantitative yield. Dimerization of **261** with $Cu(OAc)_2$ under pseudo high dilution conditions (syringe pump addition) gave mixtures of both the expected dimers and the "monomer" products resulting from intramolecular coupling of the terminal acetylenes. The monomer compound was provided in 35% yield as a yellow gum which blackened over time upon exposure to air (even when stored under nitrogen). The dimer **263** was obtained in 36% as a yellow foam.

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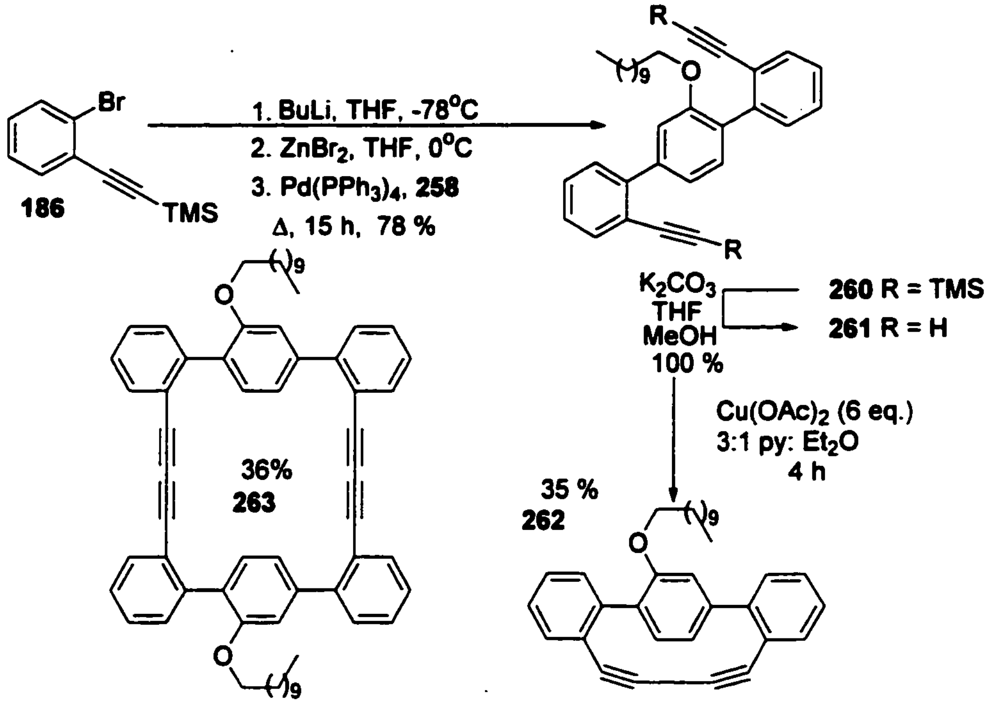
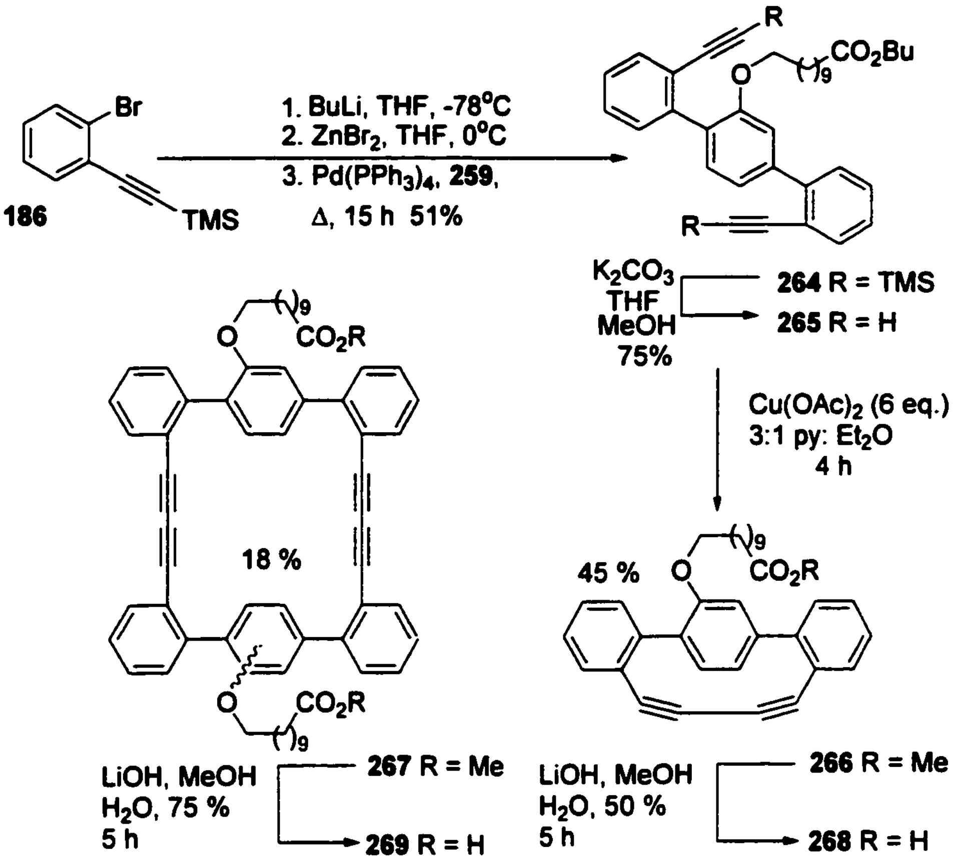
[136] Sengupta, S.; Snieckus, V. *J. Org. Chem.* **1990**, *55*, 5680.



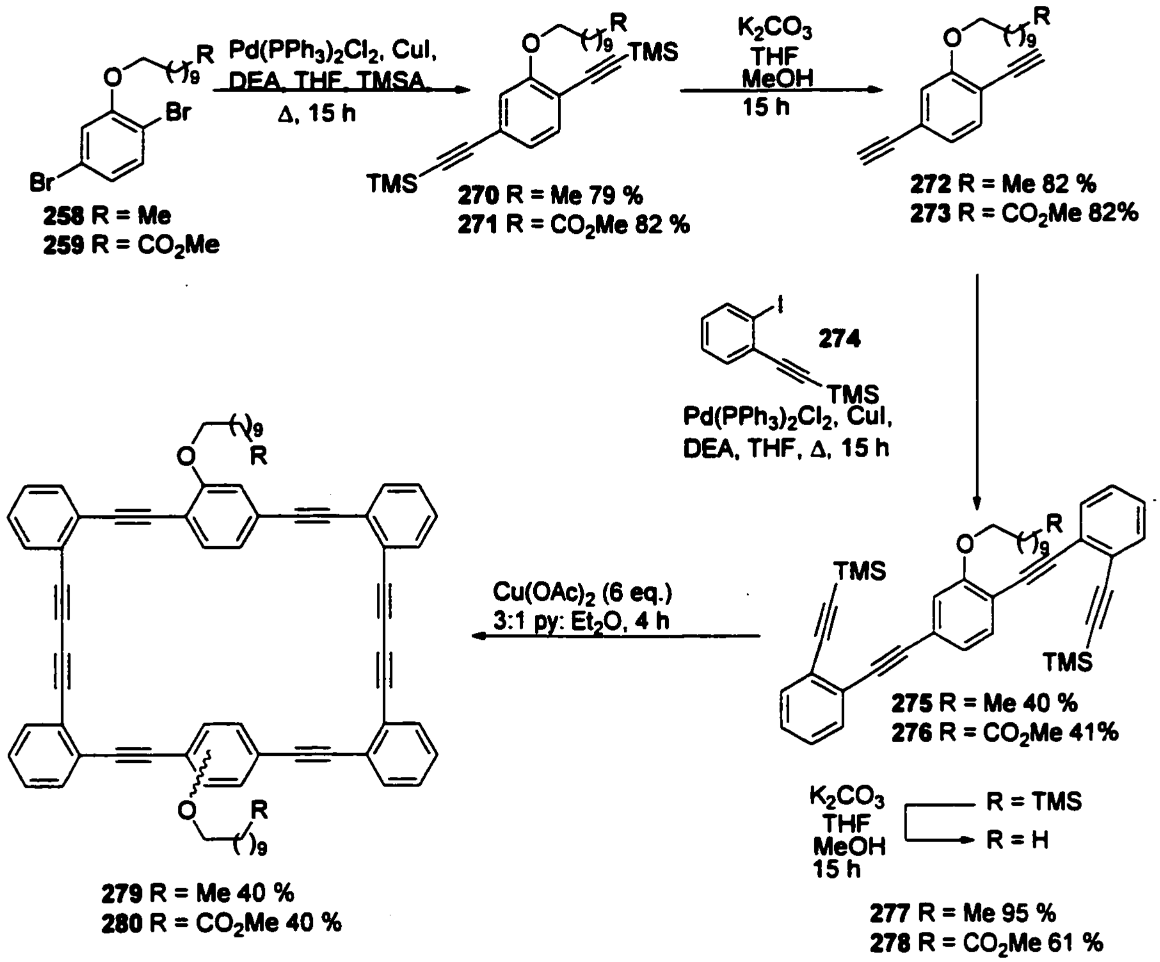
*Scheme 62. Synthesis of the potential liquid crystals, **262** and **263** bearing long alkyl chains.*

In an effort to obtain solid samples, an analogous family was constructed but the alkyl chain was altered and a methyl ester was added in place of the terminal methyl group. This group could consequently be hydrolyzed to provide polar carboxylic acid moieties for cystallization. Generation of the organozincate of (2-bromophenylethynyl)trimethylsilane followed by addition of **259** and $Pd(PPh_3)_4$ afforded the silyl protected derivative **264** in 51% yield. The transesterification may be a result of long reflux times in the presence of *n*-butylbromide, formed as a by-product from the lithium halogen exchange of **186**. A small amount of the corresponding methyl ester product was also observed by GC-MS. Deprotection of the trimethylsilyl groups gave the diacetylene **265** in quantitative yield and also resulted in a second transesterification back to original methyl ester. Dimerization of **265** with $Cu(OAc)_2$ under pseudo high dilution conditions (syringe pump addition) gave the dimer, **267** and the monomer product, **266** in 45% and 18% respectively. The monomer was again obtained as a yellow gum that blackened over time upon exposure to air or nitrogen. Hydrolysis of the methyl ester gave **268** in 50% yield and unfortunately rapidly decomposed to become a black tar over several hours. The dimer **269** was obtained as a yellow foam and when subjected to hydrolysis using LiOH in THF/MeOH/H_2O gave the corresponding diacid in 75% as an orange foam which could not be crystallized.



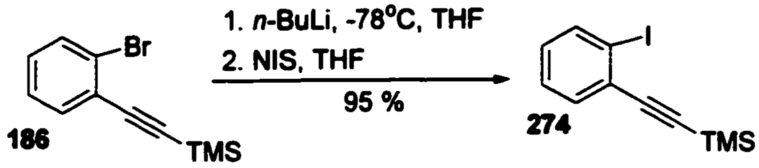
*Scheme 63. Synthesis of the potential liquid crystals, **268** and **269** bearing long functionalized alkyl chains.*

The ^1H NMR spectra obtained for the monomers indicated that the rotation of the central benzene ring had been inhibited by the formation of the butadiyne bridge. The cyclization to form the cyclophanes results in a chiral compound. Consequently, the hydrogens in the methylene group connected to the phenolic oxygen became diastereotopic and split from a well-formed triplet into two multiplets each integrating a single hydrogen. If the benzene ring were free to rotate, the chiral center within the aromatic core could racemize and the signal would remain a triplet. In addition, the ^{13}C NMR spectra obtained for the dimers indicated that a mixture of compounds had been formed. Several signals appeared as pairs of equal intensity. This is a consequence of the ability of the terminal acetylene to approach in any manner, despite the presence of the long chains, and the formation of the statistical 1:1 mixture of isomers during the dimerization. These dimers were therefore a racemic mixture of the two different regioisomers.



Scheme 64. Synthesis of the potential liquid crystals, 279 and 280 bearing long functionalized alkyl chains.

The next series of compounds was generated by insertion of an additional acetylene unit between the central and outer benzene rings of the cyclophanes. Parallel series were constructed using both **258** and **259** as cores. The additional acetylene spacers were installed by Sonogashira coupling of two molecules of TMSA and afforded the silyl protected acetylenes **270** and **271** in 82% yield. Deprotection yielded the terminal acetylenes **272** and **273** for a second Sonogashira coupling, which proceeded in 82% yield. Cross coupling with (2-bromophenylethynyl)trimethylsilane with $Pd(PPh_3)_2Cl_2$ and CuI in THF/DEA gave negligible product, even after several days at reflux. Consequently, the products **275** and **276** were obtained through coupling with the corresponding iodo derivative in 40-41% yield, prepared by treatment of (2-bromophenylethynyl)trimethylsilane with BuLi at –78 °C followed by quenching with N-iodosuccinimide (95% yield, Scheme 65).



Scheme 65. Synthesis of the iodo derivative of ***186**, **274**.*

Deprotection of the trimethylsilyl group was accomplished in 95% and 61% for **277** and **278** respectively. These were subjected to dimerization under the identical $Cu(OAc)_2$ reaction conditions used above to provide the dimers **279** and **280** in similar yields of 40-41%. Dimer **279** was a golden yellow liquid, which solidified into a brittle yellow solid when placed under vacuum, while compound **280** was obtained as a pale orange solid. Both dimers exhibited the same ^{13}C NMR effect as was observed with the standard group of cyclophanes.[137] Independent examination of the melting range of these compounds on a hot stage polarizing microscope did not reveal liquid crystal behaviour in the case of **279**. However, approximately 50% of the sample **280** melted as a clear liquid, while the remaining portion melted from 48 to 66 °C to provide a gray opaque solution typical of liquid crystal behaviour. This is likely due to the more polar regioisomer of **280** but definitive proof will require a single enantiomerically pure isomer. Unfortunately, the individual isomers could not be separated. Thus, modification of the synthetic sequence to incorporate substituents into the rigid (corner) phenyl rings would remove the potential for the formation of regioisomers.

The initial investigations show that it may be possible to use these structures as novel liquid crystals. The strength of these potential new liquid crystals is the simplicity in which they can be functionalized and altered to investigate structure-property relationships. In other words, the synthetic protocol above will also facilitate the preparation of new derivatives that may possess unusual optical properties (Figure 37). The aromatic cores can be manipulated by the addition of additional acetylene spacers that result in changing the solid-state geometries. These methods allow variation of the

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[137] Collins S.K., Yap G.P.A., and Fallis A.G. *Org. Lett.* **2000**, *20*, 3189.

chain length, alteration of the position of the alkyl groups, and modification of the acetylene spacers to control the helical conformation. In addition, the inclusion of heteroaromatic rings should permit the capture of metal ions.[138]

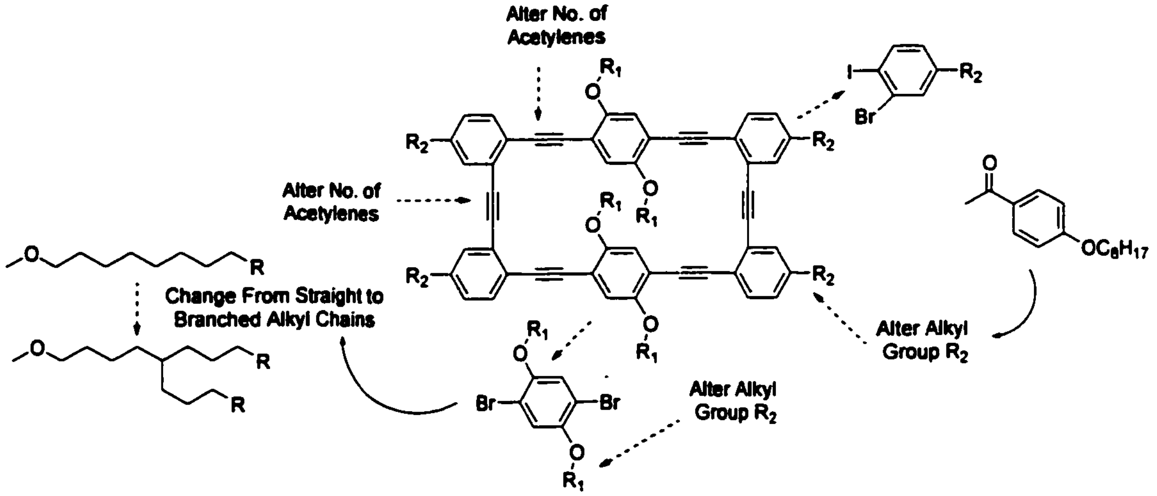

Figure 37. Various possible modifications to the potential liquid crystal phenyl-acetylene cyclophane.

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[138] (a) Baena, M. J.; Barbera, J.; Espinet, P.; Ezcurra, A.; Ros, M. B.; Serrano, J. L. *J. Am. Chem. Soc.* **1994**, *116*, 1899. (b) van Nostrum, C. F.; Picken, S. J.; Schouten, A. J.; Notle, R. J. M. *J. Am. Chem. Soc.* **1995**, *117*, 9957.

CHAPTER 5: DESIGN AND SYNTHESIS OF A NOVEL FAMILY OF 1,3,5-TRISUBSTITUTED PHENYL ACETYLENE CYCLOPHANES.

The potential for acetylenic cyclophanes to act as precursors and structural components of 3-dimensional nanostructures is well established. Studies into the potential synthesis of buckminsterfullerene using hexasubstituted cyclophanes bearing ene-yne linkages revealed that the ene-yne precursors were too unstable to undergo transition metal catalyzed chemistry at high temperatures. The goal of the following studies was to construct an acetylenic C_{60} cyclophane with the potential to form fullerene. In place of hexasubstituted benzenes, the approach was simplified by using 1,3,5-trisubstituted systems. The target intermediate, the hexasubstituted C_{30} compound, **130**, was replaced by the 1,3,5-trisubstituted molecule, **281** (Figure 38). Dimerization of the 30 carbon **281** should be routine to give our target, **282**, considering the large precedent for the formation of such systems in the literature.

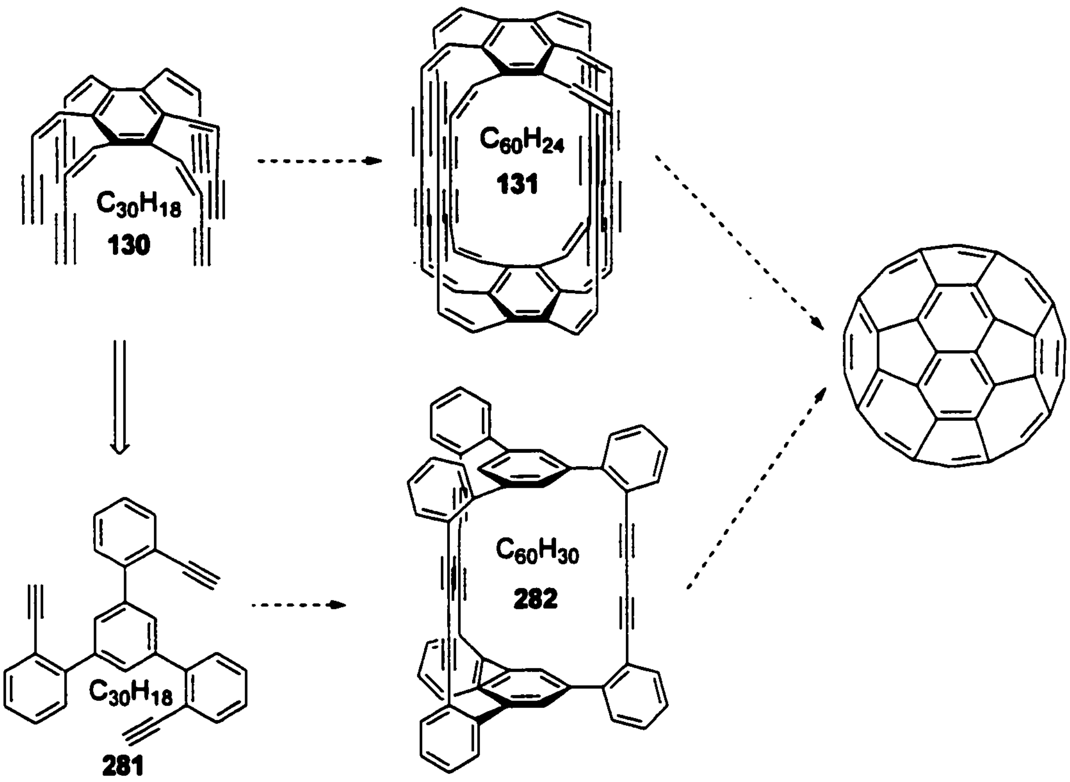


*Figure 38. Retrosynthetic analysis shows **281** to be a suitable simplified analog of **130**.*

5.1 Synthesis of 1,3,5-Trisubstituted Phenyl-yne Cyclophanes.

Oxidative dimerization of **281** should provide **282**, a cyclophane containing 60 carbon atoms and all the necessary benzene rings present in C_{60}. Pyrolysis of **282** may induce rearrangement of the carbon skeleton to C_{60}. Dimerization of the key intermediate, **281** can produce a number of other products besides the desired macrocycle (Figure 39). Incomplete oxidative dimerization may give the product, **284**, where the remaining terminal acetylenes are at opposite ends of the macrocycle and are unable to twist and complete dimerization to **282**. Consequently, **285** represents the structure that would form if the acetylenes of **284** were to thread through the structure and cyclize. The product **283** was not expected to form due to the very highly strained butadiyne linkage present within the molecule.

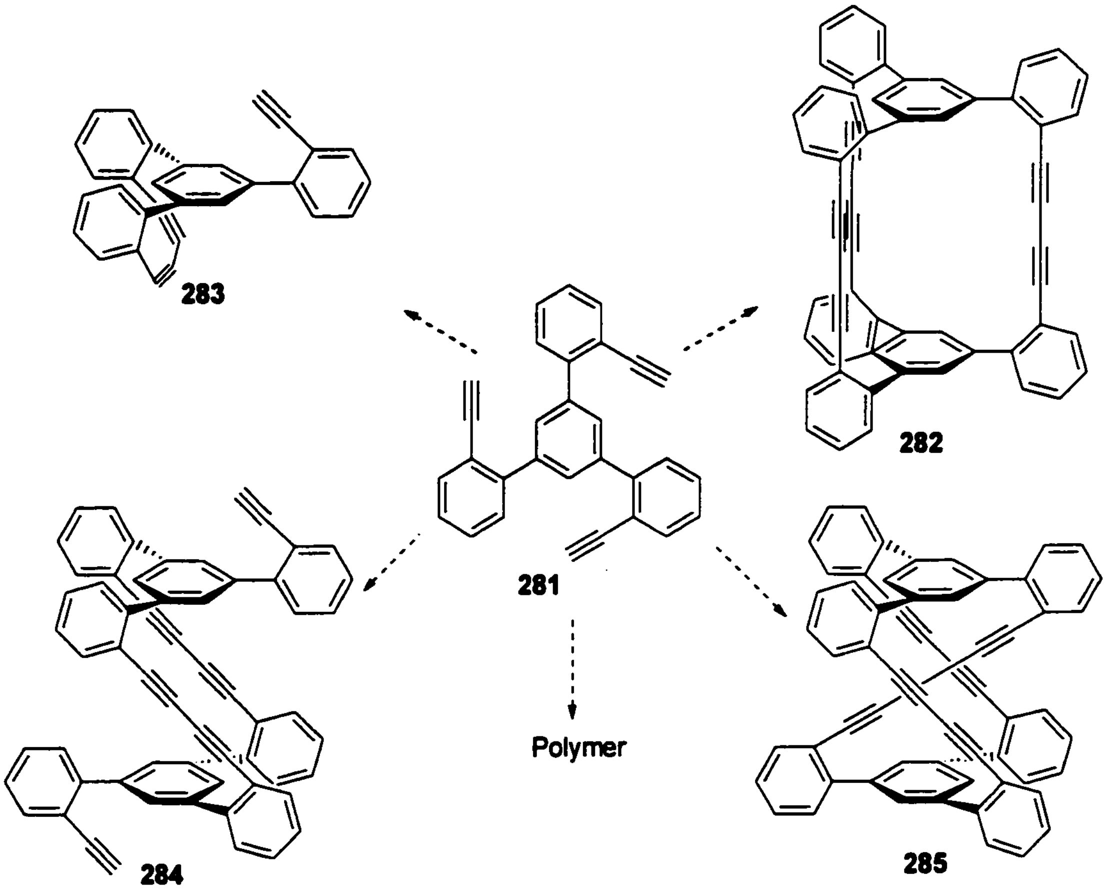
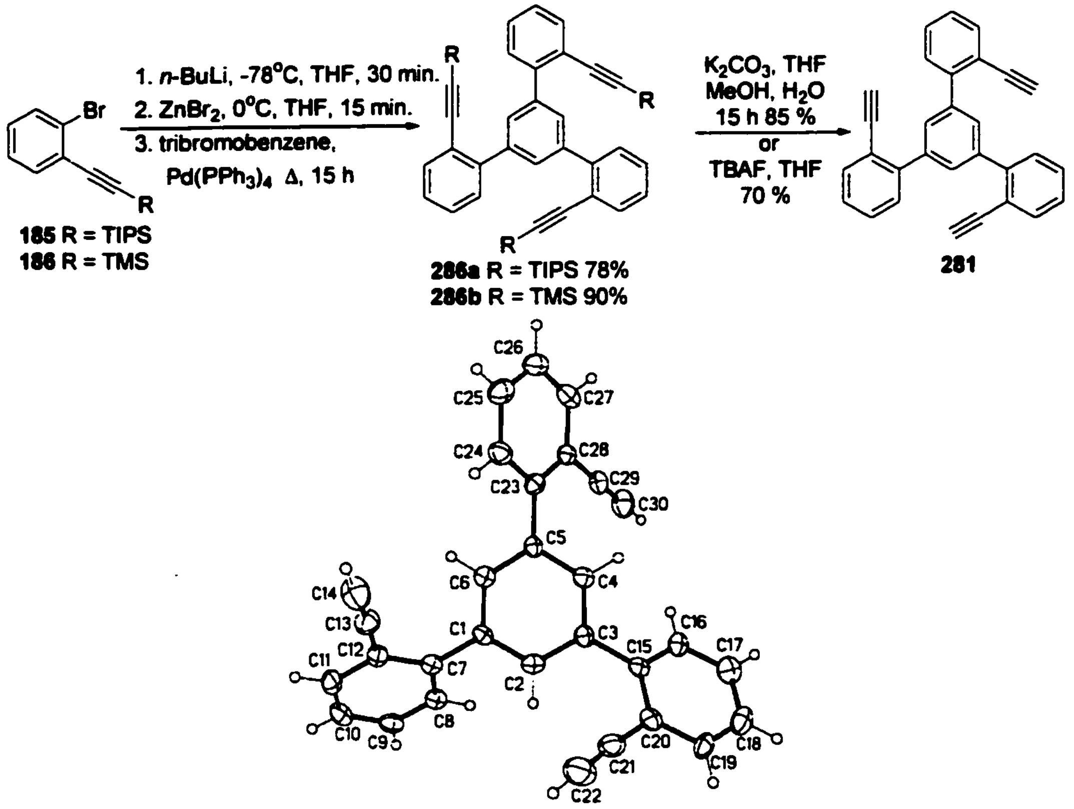


Figure 39. Possible products formed during oxidative dimerization of ***281****.*

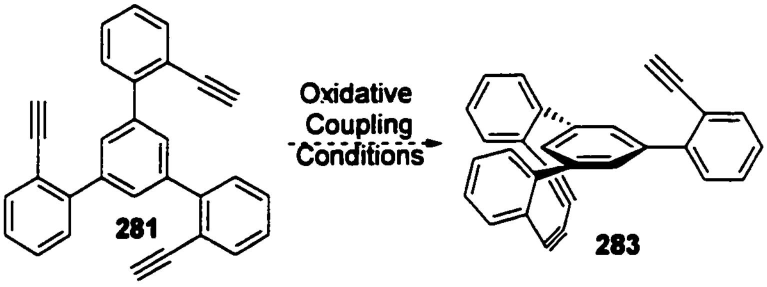
The intermediate, **281** was constructed using the established Negishi coupling protocol (Scheme 66). Transmetallation of the lithium salt of **186** with $ZnBr_2$ and coupling with 1,3,5-tribromobenzene provided **286b** in 90% yield. Subsequent deprotection with potassium carbonate in THF/MeOH gave **281** in 85% yield. The X-ray structure of **281** revealed the ethynyl groups to be in a staggered arrangement; one rests in the same plane as the central benzene ring while the others sit pointing above and below the plane (Scheme 66). A summary of the results of copper-mediated dimerization of **281** is detailed in Table 9.



*Scheme 66. Synthesis and the X-ray crystal structure of key intermediate, **281**.*

Exposure of **281** to $Cu(OAc)_2$ mediated coupling (3 equiv/acetylene) afforded the unexpected product, the highly strained cyclophane **283** in 52% yield (entry 3)! Extension of the reaction time to 3 days led to decomposition and a diminished yield of 6%. Similarly, increasing the rate of reaction by heating at reflux also reduced the yield (26%). Doubling the number of equivalents of $Cu(OAc)_2$ from 3 per acetylene to 6 per acetylene increased the yield to 62% (entry 5). Unfortunately, oxidative coupling using modified conditions produced no isolatable products (entry 6) and exposure of **281** to CuCl mediated oxidation gave only traces of **283** (entry 1). However, when palladium catalyzed coupling conditions were used, a different product was obtained in less than 10% yield. The product exhibited identical, although shifted, ^1H NMR signals compared to **283**. It was believed that the product was **284**, but to date, confirmation of the structure of the product is not possible due to the lack of spectrographic data. Thus, the major product observed from the attempted dimerization of **281** was the unusual strained cycle **283**. The cyclophane **283** was isolated as a reddish-orange powder, which decomposed slowly on standing over 3 days. There is considerable interest in strained cycloalkenes and bent polyynes, however, the growing of crystals suitable for X-ray analysis to study its butadiyne bridge of **283** was precluded by its instability.

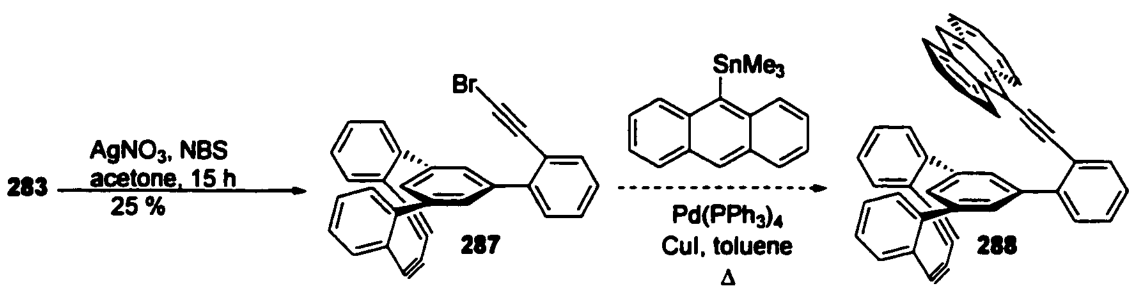
*Table 9. Oxidative Dimerization of **281**.*



Entry	Conditions	Yield (%)
1	CuCl, TMEDA, O_2 benzene, 15 h,	2
2	$Cu(OAc)_2$, (9 eq.), 3:1 py: Et_2O, 3 d, rt	6
3	$Cu(OAc)_2$ (9 eq.), 3:1 py: Et_2O, 3 h, rt	52
4	$Cu(OAc)_2$, (9 eq.), 3:1 py: Et_2O, 3 h, Δ	26
5	$Cu(OAc)_2$ (18 eq.), 3:1 py: Et_2O, 4 h, rt	62
6	$Cu(OAc)_2$ (25 eq.), CuCl (20 eq.), py, 60 °C 4 h	-
7	$Pd(PPh_3)_2Cl_2$, CuI, I_2, DIPA/THF	<10 [a]

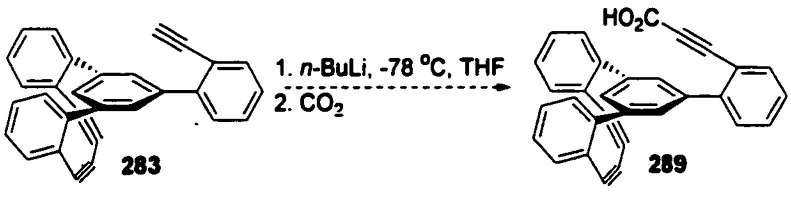
[a] Different product obtained.

Consequently, **283** was chemically modified to produce stable, crystalline derivatives. Bromination of **283** with NBS/$AgNO_3$ in acetone gave, **287** in only 25% yield. Unfortunately, Stille coupling with the known 9-(trimethylstannyl)-anthracene failed to give any of the desired product, **288**. (Scheme 67).



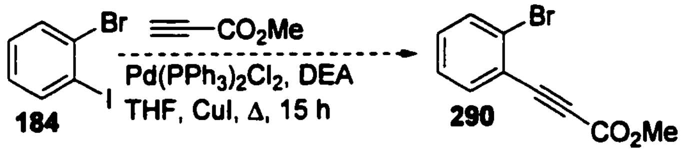
Scheme 67. Attempted construction of the ***288****.*

The success in using carboxylic acid derivatives for the X-ray structure elucidation of **203** persuaded its use for the determination of **283**. Unfortunately, treatment of **283** with 1 equivalent of n-BuLi and quenching with solid CO_2 did not give any of the desired carboxylic acid, **289** (Scheme 68). The butadiyne unit may be unstable to highly basic conditions (n-BuLi), therefore, the carboxyl functionality would have to be installed into the macrocycle at the start of the synthesis.



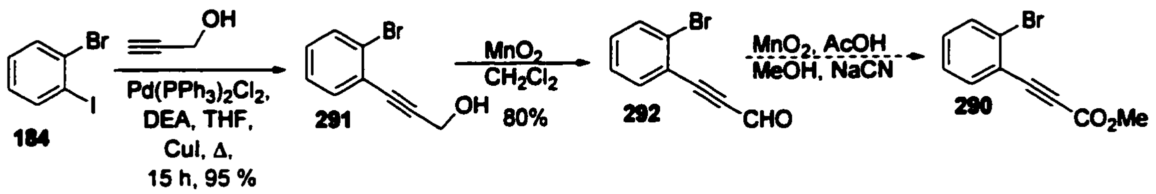
Scheme 68. Introduction of a carboxylic acid group by functionalizing ***283*** *was not possible.*

Synthesizing the precursor, **290** by direct Sonogashira coupling of 2-bromo-1-iodobenzene was not possible as methyl propiolate is too electron-withdrawing to undergo efficient palladium catalyzed cross-coupling (Scheme 69).



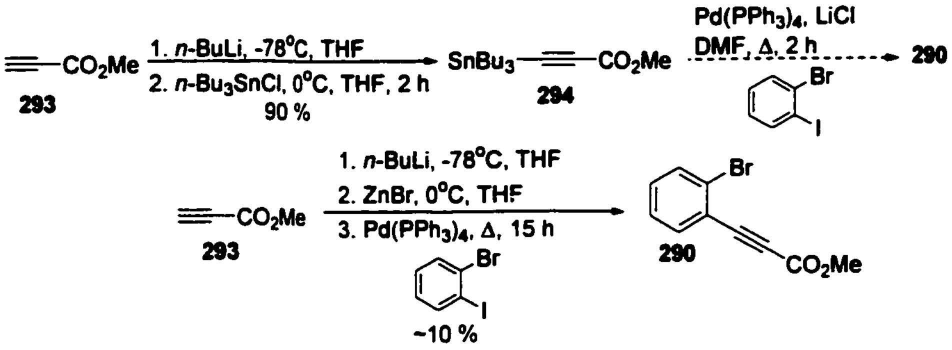
Scheme 69. Sonogashira coupling strategy for the synthesis of ***290****.*

Alternatively, propargyl alcohol can undergo Sonogashira coupling efficiently to give **291** in 95% yield (Scheme 70). The alcohol can be oxidized using MnO_2 in dichloromethane to the corresponding propargyl aldehyde but unfortunately, oxidation could not produce the desired methyl ester, **290**.



*Scheme 70. Alternative Sonogashira coupling strategy for **290**.*

Non-Sonogashira-type cross-couplings of methylpropiolate with 2-bromo-1-iodobenzene included an attempted Stille coupling with the known tri-*n*-butylstannyl acetylene,[139] **294** in the presence of $Pd(PPh_3)_4$, and LiCl in DMF but did not produce the desired product. However, deprotonation of **293** with *n*-BuLi and immediate transmetallation with $ZnBr_2$ for Negishi coupling with 2-bromo-1-iodobenzene gave the desired product in a poor 10% yield (Scheme 71). The compound **290**, was converted to its organozincate for Negishi coupling to **295**, however, **295** was recovered quantitatively (Figure 40).



Scheme 71. Organometallic cross-couplings of methyl propiolate to 2-bromo-1-iodobenzene.

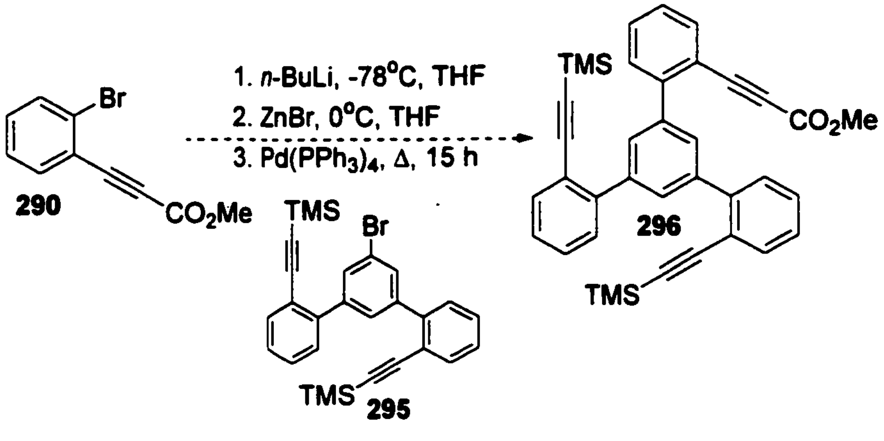


*Figure 40. Cross-coupling strategy to **296** failed.*

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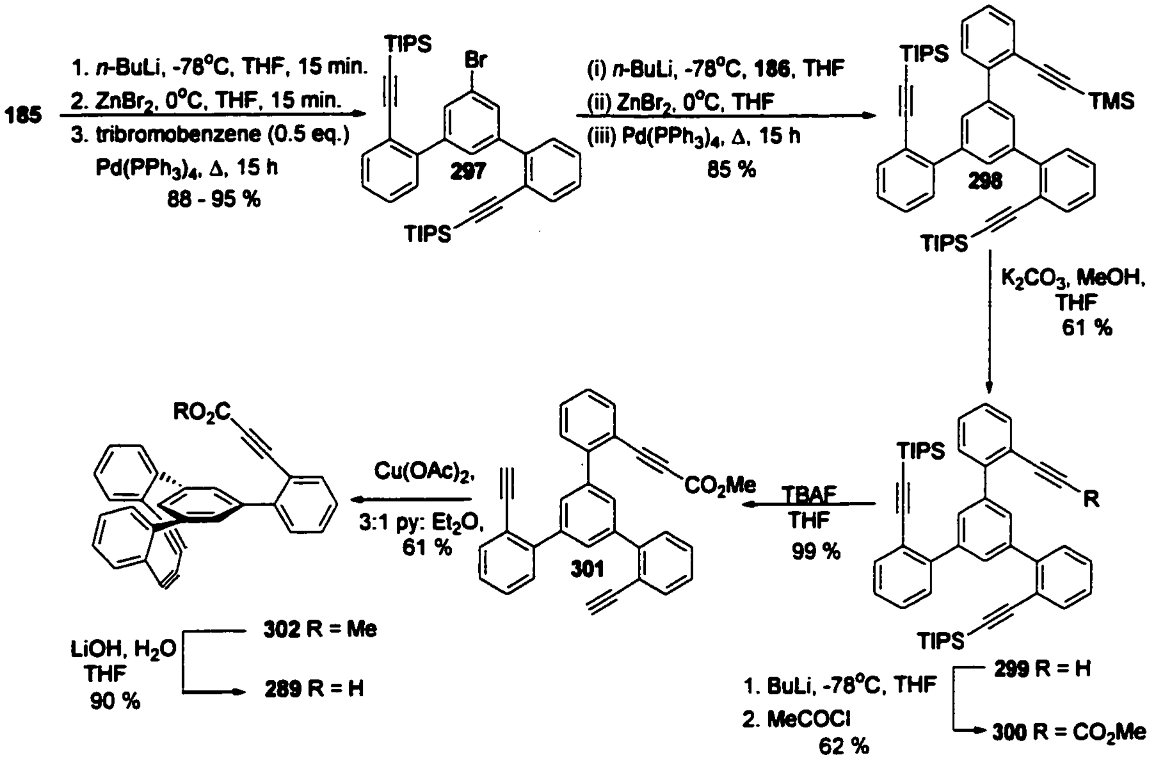
[139] (a) Sakamoto, T.; Shiga, F.; Yasuhara, A.; Uchiyama, D.; Kondo, Y.; Yamanaka, H. *Synthesis* **1992**, 746 (b) Jousseaume, B.; Villneuve, P. *Tetrahedron* **1989**, *45*, 1154.

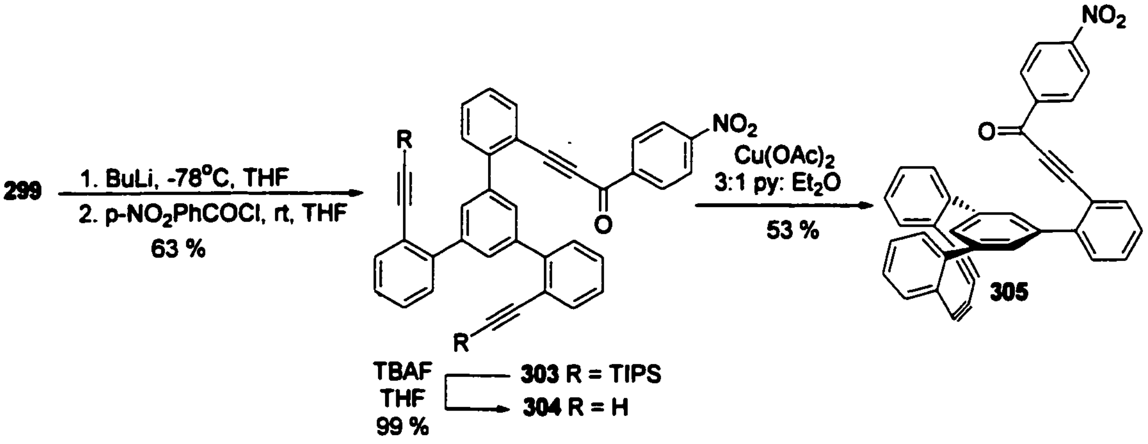
A versatile synthesis of **296** is outlined in Scheme 72 and represents a protocol for the generation of various substituted derivatives of **293**. The strategy relies on using two different trialkylsilyl protecting groups that can be removed selectively. Formation of the organozincate of **185** and coupling of two equivalents with 1,3,5-tribromobenzene give the diacetylene, **297** in 88 – 95% yield. The third acetylene is installed by a second Negishi coupling with the organozincate of **186** to give **298** in 85% yield as a thick yellow gum.



Scheme 72. Efficient strategy towards derivatives of ***283****.*

Dissolving **298** in THF/MeOH and adding potassium carbonate affects deprotection of the less table TMS silyl protecting group to give the terminal acetylene **299** in 61%. Deprotonation of **299** at –78 °C with *n*-BuLi and quenching with chloromethylformate gives the methyl ester, **300** in 62 %. The remaining TIPS protecting groups can be removed upon treatment with TBAF in THF to give the deprotected, **301** in quantitative yield. Addition of **301** *via* syringe pump to a stirred solution of Cu(OAc)$_2$ in 3:1 pyridine: diethyl ether gives the intramolecular cyclization product, **32** in moderate 61 % yield. The ester could not be crystallized but hydrolysis with LiOH in H$_2$O/THF succeeded in giving the acid, **289** in 90 % yield. Unfortunately, the acid displayed compromising solubility in aqueous and all conventional non-chlorinated and chlorinated organic solvents

Alternatively, the lithium salt of **299** could be quenched with 4-nitrobenzoyl chloride to give the aromatic derivative, **303** in 53% yield. Deprotection of the triisopropylsilyl groups occurred with TBAF to give **304** quantitatively. Cyclization by copper-mediated oxidative dimerization gave the macrocycle, **305** in 53%. Slow evaporation of dichloromethane and dichloromethane/methanol solutions of **305** gave fine hair-like needles. Unfortunately, modifying the solvents and crystallization time did not prove successful in growing crystals of a suitable size for X-ray crystallography.



Scheme 73. Synthesis of a crystalline p-*nitrobenzoyl derivative of* ***305***.

Co-crystallization of cyclophane **305** with hexafluorobenzene was attempted. The interesting quadrupole moments of benzene and its perfluoro analog have been described and complexes of this type normally occur so overlap of the two aromatic nuclei is maximized.[140] This principle was exploited for the purpose of crystal engineering to give stacked columns of *sym*-triphenethynylbenzene and *sym*-tris(perfluorophenethynyl)-benzene.[141] The perfluorophenyl-phenyl interaction has also been observed in the solution state and was used to efficiently orient large acetylenic and thiophenic cyclophane precursors for cyclization.[142] Bunz and Enkelmann have applied co-crystallization of hexafluorobenzene with the Eglinton-Galbraith dimer to elucidate its

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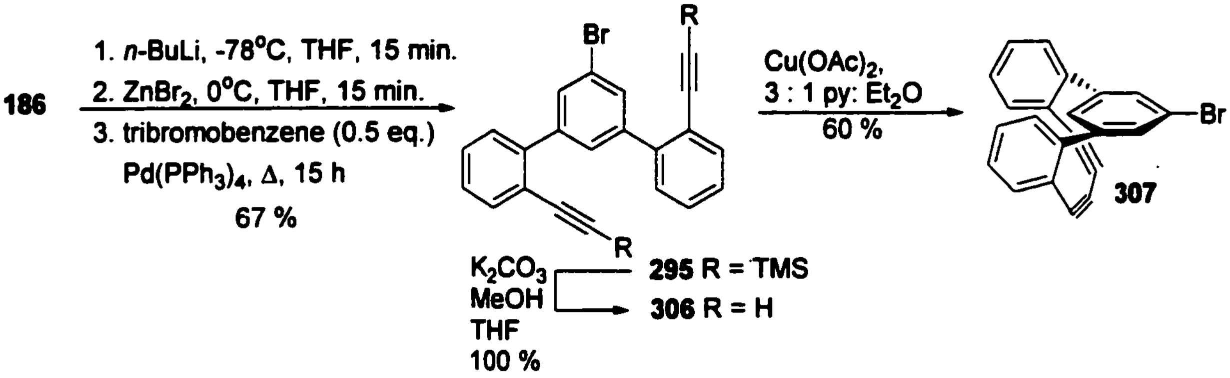
[140] Coates, G.W.; Dunn, A.R.; Henling, L.M.; Dougherty, D.A.; Grubbs, R.H. *Angew. Chem. Int. Ed. Engl.* **1997**, *36*, 248.

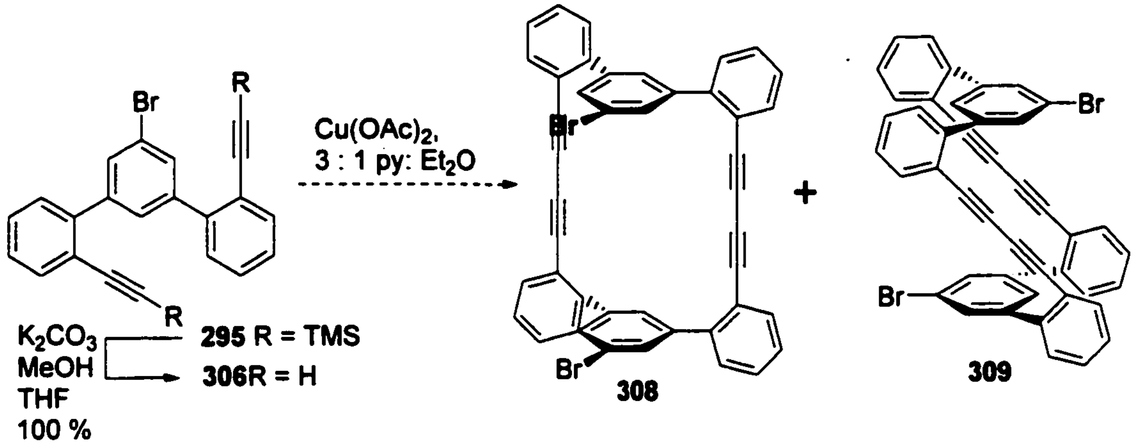
[141] Ponzini, F.; Zagha, R.; Hardcastle, K. Siegel, J.S. *Angew. Chem. Int. Ed. Engl.* **2000**, *39*, 2323.

[142] Marsella, M.J.; Wang, Z.-Q.; Reid, R.J.; Yoon, K. *Org. Lett.* **2001**, *3*, 885.

solid state structure.[143] They observed that the co-crystallization with hexafluorobenzene produced stable crystals at room temperature as platelets while the dimer normally produced thin unstable needles. The X-ray crystal packing revealed that the hexafluorobenzene rings sat above and below the plane of the dehydroannulene ring and are not the expected benzenoid portions of the molecule. Thus, the instability of the dimer in the crystal state is due to the nature of the crystal packing and not the strained butadiyne bridges. The intercalation of hexafluorobenzene acts as a spacer to separate the molecules form one another in the crystal lattice.



Scheme 74. Construction of the crystalline bromide derivative, 307.



Scheme 75. Intermolecular dimerization products were not observed in the cyclization of 306.

Surprisingly, compound, **307**, containing an aromatic bromine substituent, resulted in crystallization and structure elucidation. The structure was constructed from **186** in a parallel manner to **283** (Scheme 74). Coupling of two equivalents of the organozincate of **186** with 1,3,5-tribromobenzene gave **295** in 67%. Deprotection of the

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[143] Bunz, U.H.F.; Enkelmann, V. *Chem. Eur. J.* **1999**, *5*, 263.

TMS silyl protecting groups gave the terminal diacetylene, **306** quantitatively. Oxidative dimerization of **306**, gave exclusively the strained undecadiyne **307** in 60% yield, corresponding to the intramolecular cyclization observed previously. No products resulting from intermolecular dimerization were observed in the reaction mixture (Scheme 75).

Crystals of **307** were obtained as thin needles from the slow evaporation of a dichloromethane solution. This process created a dark film from competitive decomposition that was removed from the crystals prior to analysis. The crystal structure (Figure 41) revealed the strained nature of the acetylene linkages within the cyclophane. The phenyl units remain free of any distortion and adopt an orientation that forced the acetylene linkages below the plane of the central benzene ring. This conformation places the *para* hydrogen atom just above the diacetylene bridge. The ^1H NMR signal for this hydrogen in **306** appears as a triplet at 7.89 ppm, but experiences a significant deshielding effect upon cyclization to **307**, shifting to 8.25 ppm. This hydrogen occupied the space between the bridge and the central benzene ring and thus no cavity is evident, despite the appearance of the diagram.

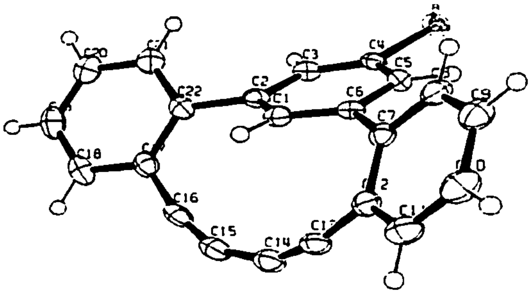

Figure 41. X-ray crystal structure of 307.

The adjacent acetylene units are distorted, as a consequence of the cyclization, and deviate ~16-26° from planarity. The bond angles of the triple bonds are C_{12}-C_{13}-C_{14} 164.1° and, C_{14}-C_{15}-C_{16} 153.4°. In most cases, these bond angle distortions are greater than those reported for early[66,67,68] and recent diyne systems [144,71,72]. Additional strained

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[144] Baldwin, K. P.; Matzger, A. J.; Scheiman, D. A.; Tessier, C. A.; Vollhardt, K. P. C; Youngs, W. J *Synlett* **1995**, 288.

diynes systems have acetylene bond angles of 165.8°,[145] 172.8°,[146] 168.0°,[147] and 164.5.[148]

The ^{13}C NMR chemical shifts of the *sp* carbons also experience a deshielding due to ring strain. The chemical shifts of the acetylenic carbons in **307** are 87.1 and 107.4 ppm. These values are consistent with the trend described by Tykwinski and co-workers[18] for strain in butadiyne bridges as the chemical shifts for the *sp* carbons in **109** (n=1) appeared at 86.5 and 108.1 ppm for bond angles of 159.4° and 156.2°.

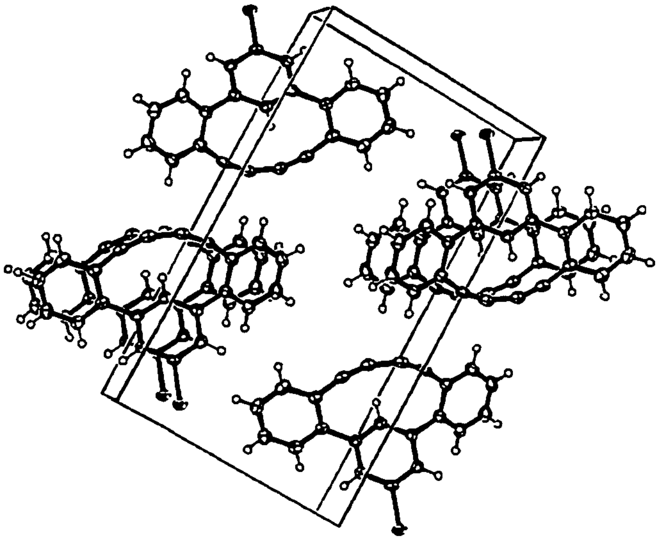

*Figure 42. The X-ray packing diagram reveals that **307** sit on top of one another as a stack of coins.*

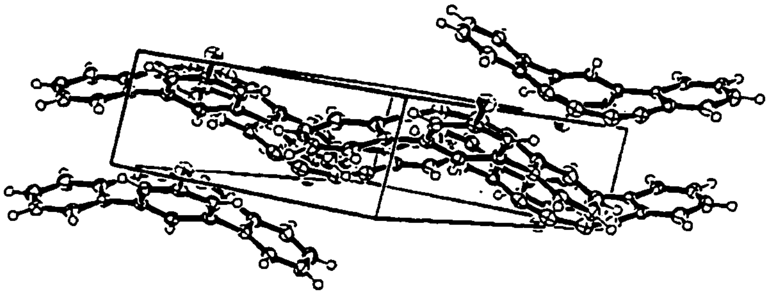

*Figure 43. The X-ray packing diagram displays the concave conformation of **307**.*

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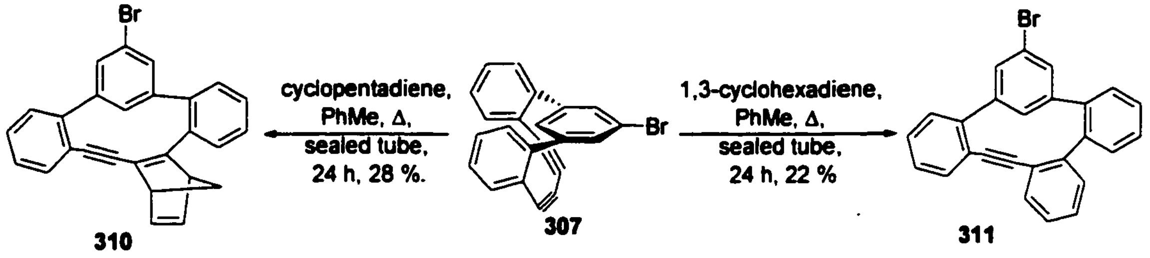
[145] Zhou, Q.; Carroll, P. J.; Swager, T. M. *J. Org. Chem.* **1994**, *59*, 1294.
[146] Ueda, H.; Katayama, C.; Tanaka, J. *Bull. Chem. Soc. Jpn.* **1981**, *54*, 891.
[147] Ohkita, M.; Ando, K.; Suzuki, Tsuji, T. *J. Org. Chem.* **2000**, *65*, 4385
[148] Anthony, J.; Knobler, C. B.; Diederich, F. *Angew. Chem. Int. Ed.* **1993**, *32*, 406.

The crystal packing of the **307** reveals some the molecules sit on top of one another as in a stack of coins with the butadiyne bridge units overlapping (Figure 42). This is unusual in that other molecules reported by Bunz and Fallis have shown that the molecules having strained annulene cores tend to overlap these portions of the molecule with the benzenoid moiety of another. The concave shape of the molecule can also be seen in looking at the lattice from a side view (Figure 43). The intimate packing and proximity of the reactive bridges may aid in polymerization of the molecule and contribute to its observed instability in the crystal state.

In most circumstances, very reactive inverse demand dienes such as cyclopentadienones are required to effect [4+2] cycloadditions with diphenylacetylene and 1,4-diphenyldibutadiynes.[149] In the case of the cyclic 'diphenyldibutynyl' system **307**, the exceptionally strained nature of the butadiyne bridge was confirmed *via* standard Diels-Alder reactions with 1,3-cyclohexadiene and cyclopentadiene (Scheme 76).



Scheme 76. Exploring the dienophilic character of the butadiyne bridge of 307.

The cyclophane **307** underwent cycloaddition with cyclopentadiene (sealed tube, 120 °C) to give the bicycloadduct, **310**, in 28% yield. The low yield was attributed to the thermal instability of **307**. The cycloaddition reaction was not observed at room temperature (21 °C) nor after 24 hours at reflux (110 °C) at atmospheric pressure. A second addition was not detected, as the first cycloaddition relieves the strain in the butadiyne unit and reinstates an approximately linear bond angle geometry in the remaining triple bond. In a parallel fashion, **307** underwent initial cycloaddition with 1,3-cyclohexadiene, followed by thermal expulsion of ethylene to give the interesting acetylenic-tetraphenyl product **311**. These results established the olefinic nature of this distorted triple bond and its rare dienophilic characteristics.

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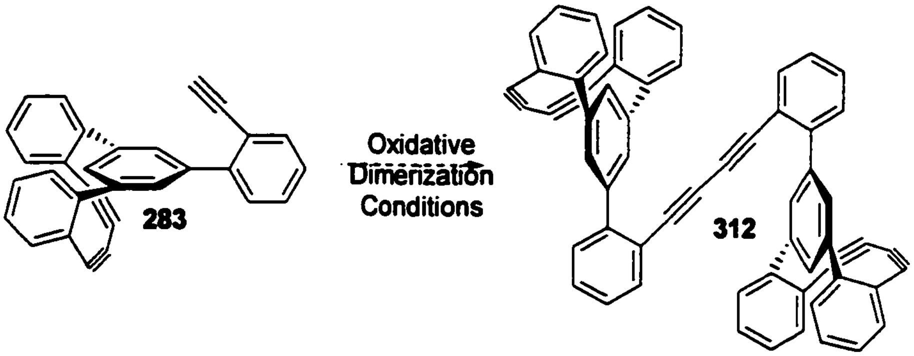
[149] Tovar, J. D.; Jux, N.; Jarrosson, T.; Khan, S. I.; Rubin, Y. *J. Org. Chem.* **1997**, *62*, 3432.

It was also of interest that upon dimerization of **281** the strained **283** formed preferentially but coupling of the remaining terminal acetylene did not occur. Considering the rate of dimerization of acetylenes approaches diffusion-controlled limits, its surprising that the remaining terminal acetylene of **283** does not self-condense. Attempts at dimerizing isolated **283** in a separate chemical step are outlined in Table 10. Despite a variety of reaction conditions it was not possible to isolate the product, **312**, and only recovered starting material was recovered, which may suggest that the acetylene is sitting in a steric environment that makes it unavailable for reaction.

Table 10. Attempted dimerization experiments of ***283****.*

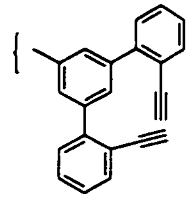
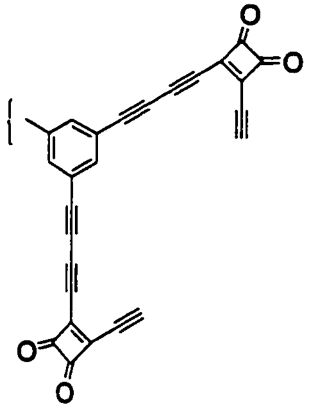
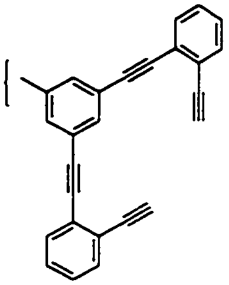
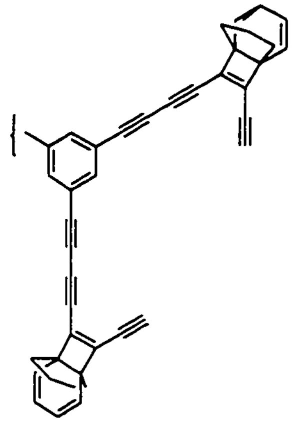
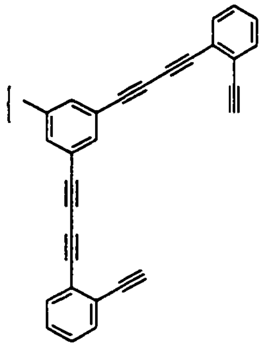
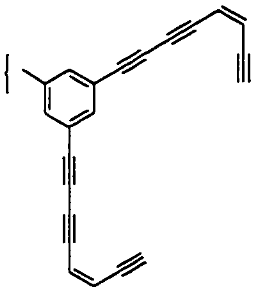


Entry	Conditions	Yield Recovered **283** (%)
1	$Cu(OAc)_2$ (9 eq.), 3:1 py: Et_2O, rt, 4 h	66
2	$Cu(OAc)_2$ (9eq.), 3:1 py: Et_2O, rt, 24 h	50
3	$Cu(OAc)_2$ (9 eq.), 3:1 py: Et_2O, 30 °C, 3 d	10
4	CuCl, TMEDA, O_2, benzene, 15 h	-

5.2 Extended 1,3,5-Trisubstituted Cyclophanes. Intra- vs. Intermolecular Cyclization: The Role of Termini Separation.

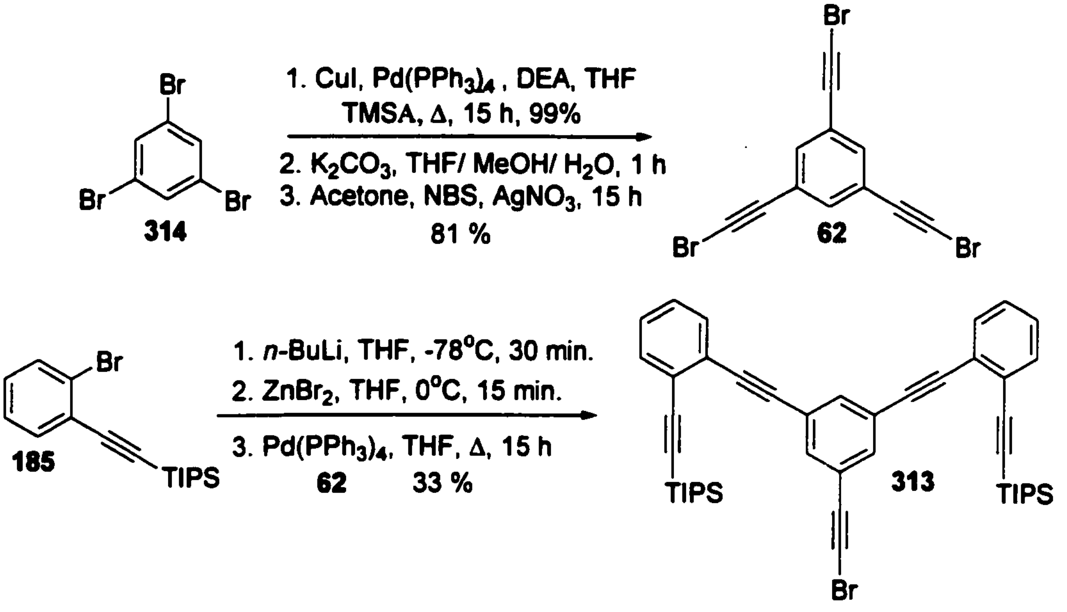
The observation that exposure to copper-mediated oxidative dimerization conditions yields the strained molecule **283** from intramolecular cyclization is similar to the observation that similar intramolecular cyclization were observed for 1,4-disubstitted phenyl acetylene cyclophanes. It was decided to study the competitive intramolecular and intermolecular reactions with regards to the intramolecular distance between the relative acetylene termini.

Table 11. Intramolecular Distances of Terminal Acetylenes in Various 1,3,5-trisubstituted Cyclophane Precursors.

Entry	Cyclophane Precursor	Separation (Å)	Entry	Cyclophane Precursor	Separation (Å)
1		1.34	4		9.19
2		3.45	5		9.22
3		7.81	6		7.76

Examination of the relevant literature, indicated that for successful dimerization of related multibridged macrocycles, all compounds possessed two acetylene units between the central benzene ring and the outer unsaturated unit. In these cases, the authors did not report any competing intramolecular coupling nor the observation of by-products resulting from such coupling. It is again theorized that these extra acetylene units place the reactive acetylene termini at sufficient distance to inhibit intramolecular coupling. This distance must be sufficient to render intramolecular coupling improbable since cyclophanes **283** and **307** suggest that, when possible, intramolecular coupling predominates even when the products prove to be highly strained.

Table 11 summarizes the calculated[150] separations between two terminal acetylene units in the related precursors. For example, **281** (entry 1) and **313** (entry 2) exhibit intramolecular distances of approximately 1.34 and 3.45 Å. However, the addition of another alkyne unit adjacent to the central benzene ring extends the intramolecular distance to 7.81 Å (entry 3). Consequently, entries 4,[55] 5[53] and 6[52] all represent molecules previously synthesized, where the intramolecular distance between reactive acetylenes ranges between 7.76 and 9.22 Å and thus no intramolecular coupling was observed.



Scheme 77. Synthetic strategies towards the extended 1,3,5-trisubstituted cyclophane precursor, 313.

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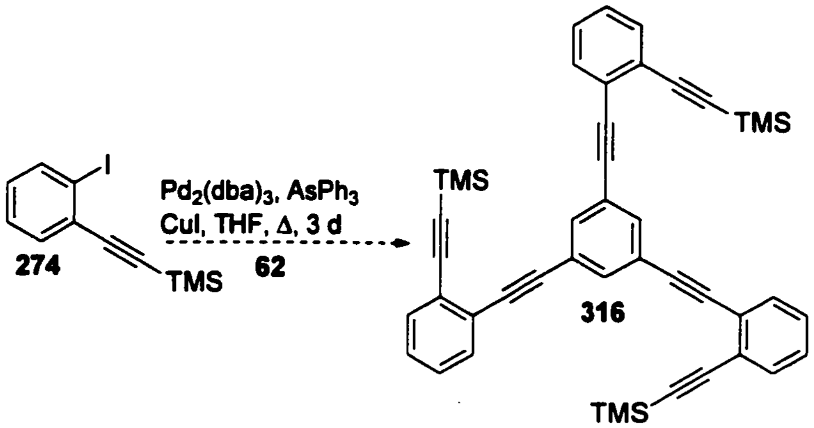
[150] Calculations were performed using CAChe Editor v3.7 and CAChe Mechanics v3.7 systems. Structures were optimized by MM2 parameters.

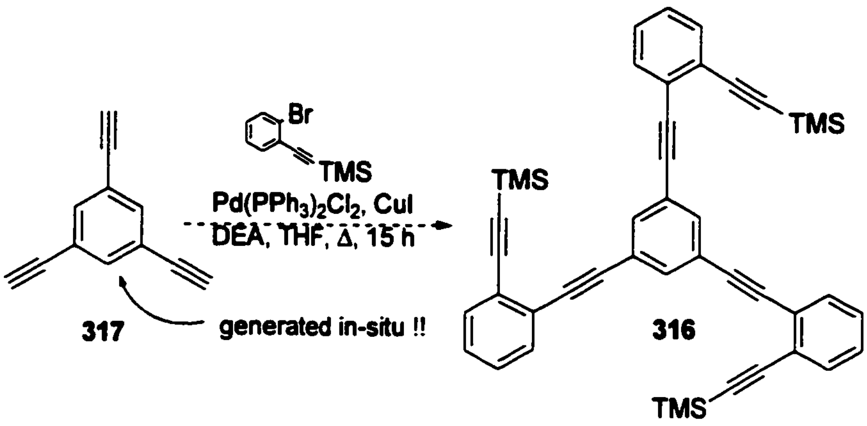
The cyclophane, **313** bearing additional acetylene spacers between the outer phenyl rings and the central benzene rings was constructed. The central precursor was easily obtained through Sonogashira coupling of three equivalents of TMSA to 1,3,5-tribromobenzene to give the triacetylene in quantitative yield (Scheme 77). Subsequent deprotection and bromination of the terminal acetylenes gave the product, **62** in 81% overall for the two steps. Unfortunately, Negishi coupling of the organozincate of **185** with **62** failed to give any of the desired tri-coupled product and instead gave a low yield (33%) of the di-coupled product, **315**.

A subsequent coupling was investigated to couple the iodo derivative **274** to **62** in the presence of $Pd_2(dba)_3$, CuI and $AsPh_3$ in refluxing THF but no product was obtained and the starting materials were recovered. (Scheme 78).



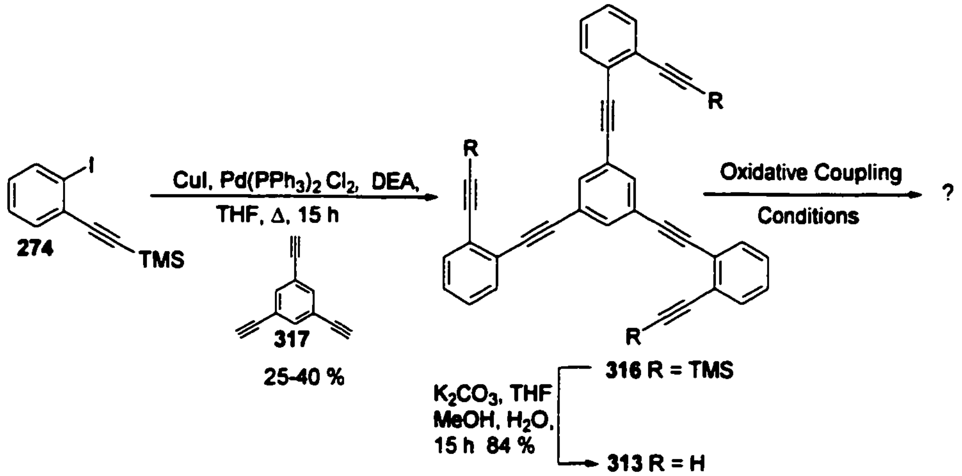
Scheme 78. Failed Stille coupling strategy to 316.

The failed cross-couplings were due to the lack of reactivity of the tribromide, **62**. Instead, 1,3,5-triethynylbenzene, **317**, was generated *in situ* by adding 1,3,5-(trimethylsilylethynyl)benzene and **186**, and powdered potassium carbonate with the palladium and copper catalysts (Scheme 79). Similar polysubstituted ethynylbenzenes such as 1,2,4,5-tetraethynylbenzene has been known to be explosive upon scratching. Unfortunately, this strategy also failed.



Scheme 79. Sonogashira strategy for the synthesis of 316.

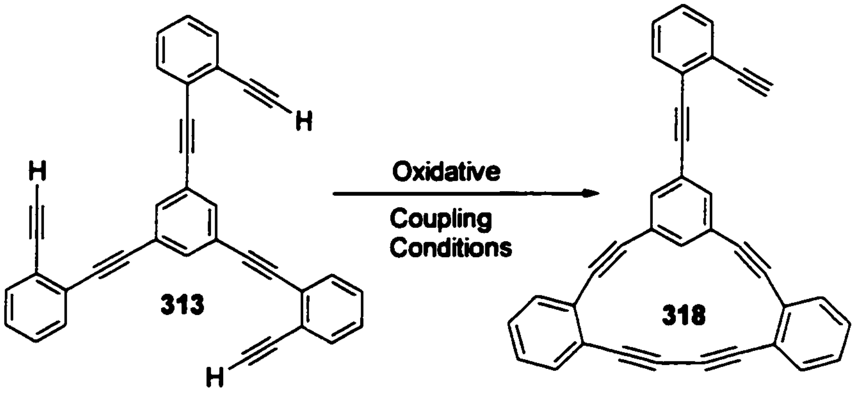
The triactylene, **317** was isolated as an off-white solid and cross-coupled to 3 equivalents of **274** to give the desired product in 25-40% yield. The trimethylsilyl protecting groups could be desilylated under basic conditions to give **313** in 84% yield (Scheme 80).



Scheme 80. Synthesis of 313 via *Sonogashira coupling of iodo arenes and terminal acetylenes.*

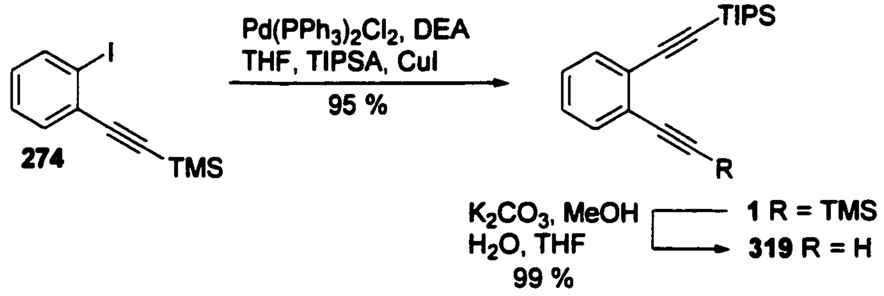
Upon investigation of the dimerization of **313** under a variety of copper-mediated oxidative protocols, it was observed that only the product arising from intramolecular coupling was isolated (Table 12). Unfortunately, this could not be proven unambiguously as the molecule did not give satisfactory mass spectrographic analysis by electron impact, FAB and electrospray techniques and all attempts to crystallize the product failed.

Table 12. Copper-mediated dimerization of 313.



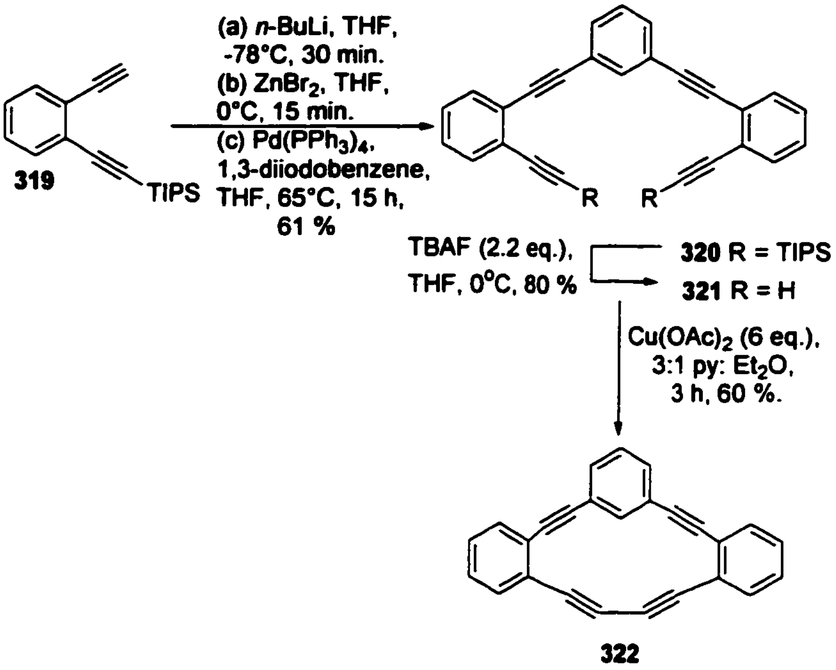
Entry	Conditions	Yield (%)
1	$Cu(OAc)_2$, 9 eq., py: Et_2O 3: 1, 3 h, Δ	6
2	$Cu(OAc)_2$, 9 eq., py: Et_2O 3: 1, 3 h, rt	15
3	$Cu(OAc)_2$, 9 eq., py: Et_2O 3: 1, 3 d, rt	10

As a result, the 1,3-disubstituted cyclophane **322** was constructed as a model. The appropriate precursor, **319** was synthesized by Sonogashira coupling of TIPSA to **274** followed by selective deprotection of the trimethylsilyl group with potassium carbonate.

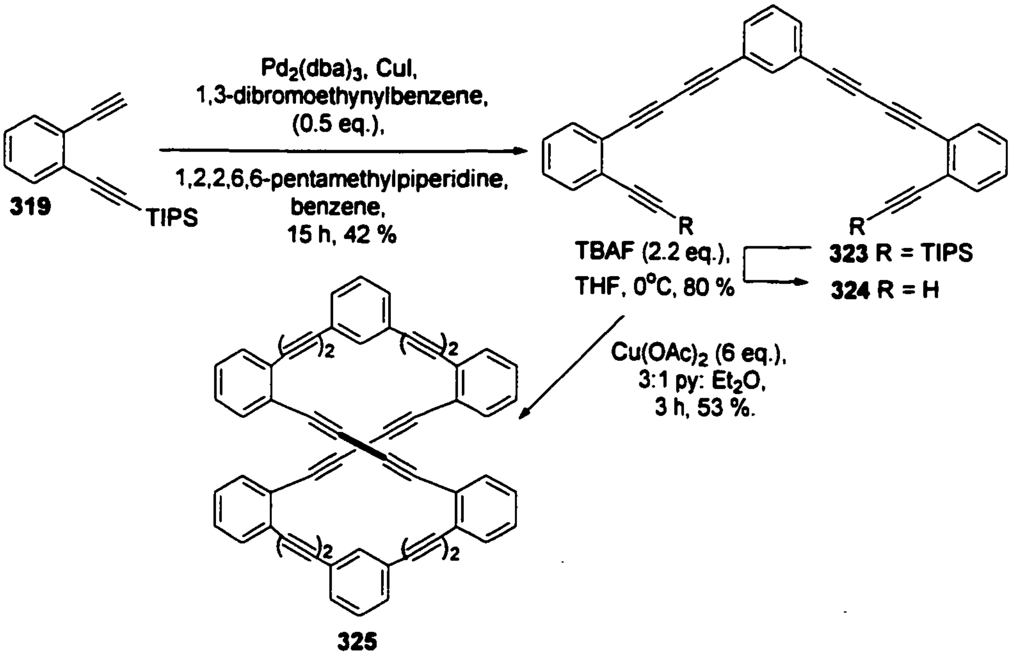


Scheme 81. Selective deprotection of the trimethylsilyl groups allows preparation of 319.

Generation of the organozincate of **319** and subsequent coupling to 1,3-diiodobenzene generated the compound precursor, **320** in 61% yield. Desilylation with fluoride ion (TBAF) gave the target **321** in 80% yield. Dimerization upon addition by syringe pump to a copper acetate solution gave only the product resulting from intramolecular cyclization, **322**.



Scheme 82. Intramolecular cyclization of the extended 321 results in formation of strained cyclophanes.



Scheme 83. Dimerization of 324 should yield primarily the product 325 resulting from intermolecular coupling.

Accordingly, the acetylenic precursor, **324**, a derivative of **321** in which two alkynes units are connected to the central benzene ring was synthesized and was expected to give the dimeric product **325** upon copper mediated oxidation (Scheme 83). Coupling of two equivalents of **319** to 1,3-(dibromoethynyl)benzene produced **323** in 42% yield. Desilylation of **323** gave the 80% yield to give the terminal acetylene, **324**, for oxidative dimerization. Upon exposure to copper acetate solution, a single product was obtained. This material was sparingly soluble in all organic solvents and the majority of the product was lost during chromatographic purification. Consequently, to date the identity of the product **325** remains in question. However, organic solvent soluble products corresponding to intramolecular cyclization products were not observed.

5.3 Sequential Coupling Routes to 1,3,5-Trisubstituted Cyclophanes.

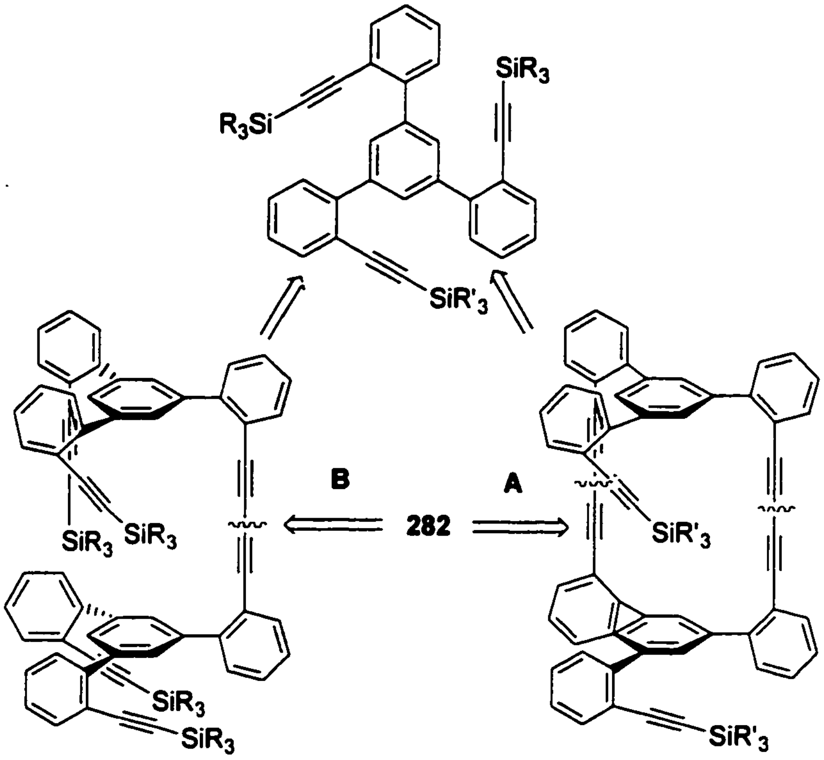
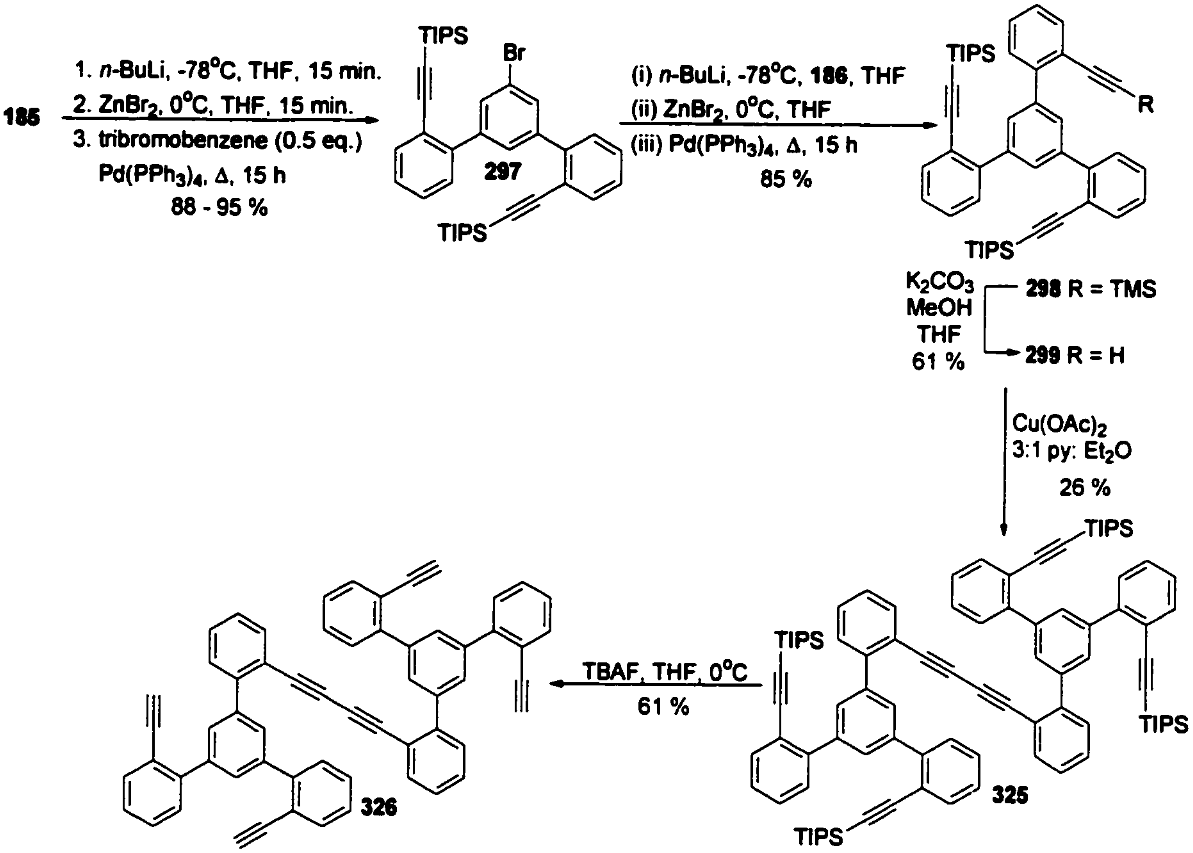


*Figure 44. Retrosynthetic analysis reveals two independent pathways for a sequential coupling approach to **282**.*

With the goal of synthesizing the 60-carbon cyclophane, **282** in mind, a sequential coupling approach was envisioned as an alternative strategy. The retrosynthetic outline (Figure 44) involves selectively forming one or two of the butadiyne bridges in two independent chemical steps. The pathway "A" concerns forming two of the linkages before final closure of the third bridge. This route is not possible due to the competing intramolecular cyclization to form strained cyclophanes. The second pathway "B" would involve an intramolecular closure of the two final bridges after initial formation of the first butadiyne linkage. This intramolecular reaction is expected to compete with the previously observed intramolecular cyclization to formed strained cyclophanes (Figure 44).

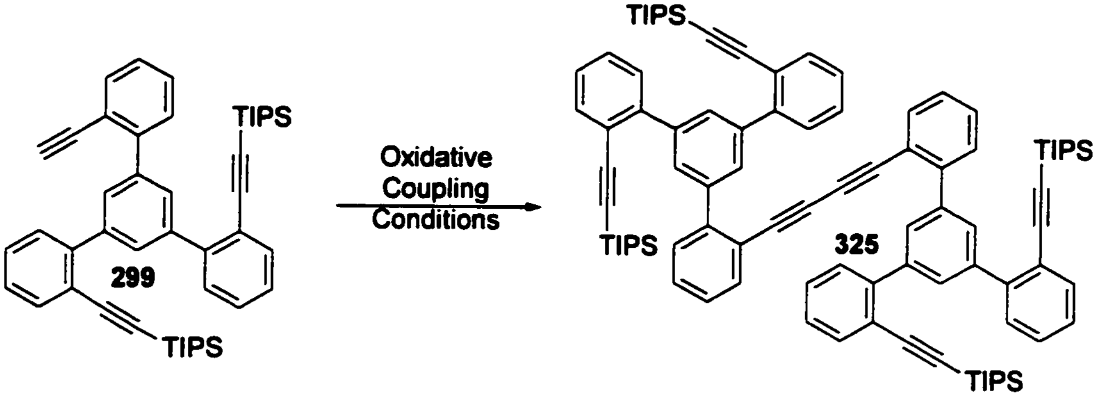
The synthesis of the precursor **326** is analogous to that used to construct the carboxylic acid derivative, **289**. Negishi coupling of two equivalents of the organozincate of **185** to 1,3,5-tribromobenzene occurs in 88 - 95% yield. A second coupling with the corresponding organozincate of **186** installs the third phenyl acetylene unit. Selective deprotection of **298** to give **299** occurs in moderate 61% yield, presumably due to the steric hindrance of the two adjacent triisopropylsilyl groups. The steric encumbrance may also be responsible for the difficulty observed in dimerizing **299** to generate the first of the butadiyne bridges necessary for **282**.



Scheme 84. Construction of the precursor, 326 for sequential coupling to 282.

Under standard Eglinton coupling conditions, **299** dimerized in just 26% yield (Table 13, entry 1). However, tripling the concentration of the reaction mixture from 1.5 mM to 4.5 mM increased the yield to 56% (entry 2). Increasing the concentration tenfold to 15 mM results in similar yields as observed with concentrations of 4.5 mM (entry 3). Interestingly, changing the copper catalyst system and keeping the concentration at a relatively dilute 1.5 mM gave the best yields of **325** at 72% (entry 4).

Table 13. Dimerization of ***299*** *at various concentrations.*

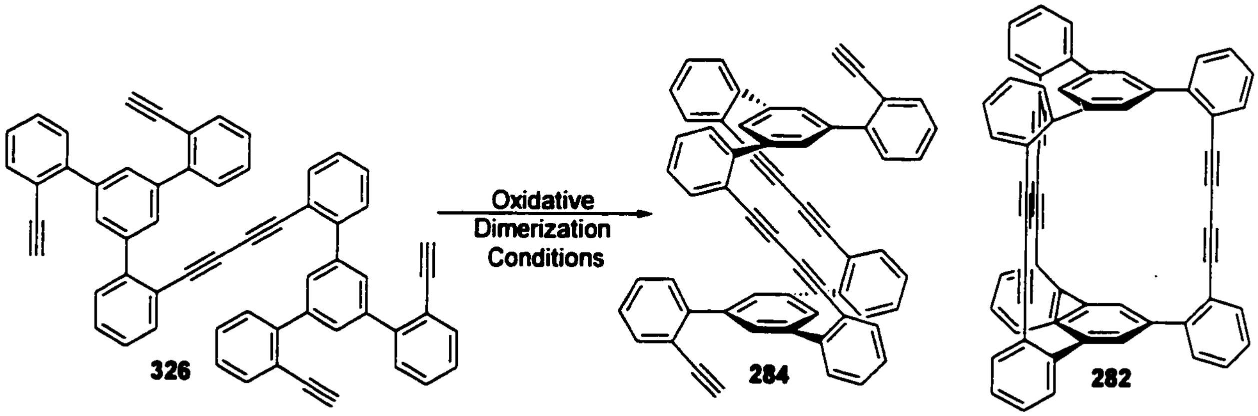


Entry	Conditions	Concentration (mM)	Yield (%)
1	Cu(OAc)$_2$, (3 eq.), 3:1 py: Et$_2$O, 4 h, rt	1.5	26
2	Cu(OAc)$_2$, (3 eq.), 3:1 py: Et$_2$O, 4 h, rt	4.5	56
3	Cu(OAc)$_2$, (3 eq.), 3:1 py: Et$_2$O, 4 h, rt	15	41-62
4	CuCl, TMEDA, benzene, 4 h, rt	1.5	72

A four-fold desilylation of **325** with TBAF at 0 °C gave the tetraethynyl precursor, **326** as a yellow thick gum in 61% yield. The results of the standard addition of **326** by syringe pump to a stirring solution of copper or palladium catalysts are described in Table 14. Addition of **326** to Cu(OAc)$_2$ at a concentration of 1.5 mM in 3:1 pyridine: diethyl ether for 3 hours gave only traces of products (Table 14, entry 1). Increasing the concentration three-fold produced traces of the desired product but gave a small yield of **284** that exhibited marginal stability in solution. This product had a similar ^{1}H NMR spectra to the product obtained during the dimerization of **281** under Pd-catalyzed conditions (Table 9, entry 7) and was assigned to be **284**. Similarly, the traces of the second product obtained produced a singlet in the ^{1}H NMR spectra at ~8.5 ppm.

The product was proposed to be **282**, due to the assumption that the macrocycle would have 6 equivalent hydrogen atoms around the central benzene rings. Using Hay coupling conditions did not produce any change in the observed yields (entry 3) and palladium coupling conditions resulted in a very slow reaction resulting in extensive decomposition of the starting material (entry 4).

*Table 14. Sequential coupling to produce **284** and **282**.*



Entry	Conditions	Concentration (mM)	Yield **284** (%)	Yield **282** (%)
1	$Cu(OAc)_2$, (6 eq.), 3:1 py: Et_2O, 4 h, rt	1.5	Trace	Trace
2	$Cu(OAc)_2$, (6 eq.), 3:1 py: Et_2O, 4 h, rt	4.5	10	Trace
3	CuCl, TMEDA, benzene, 15 h, rt	4.5	5	Trace
4	$Pd(PPh_3)_2Cl_2$, CuI, I_2, DIPA/THF	1.5	-	-

The fact that more of **284** was produced in the dimerization of **326** is not surprising. Since the couplings should occur sequentially, it is unlikely that **282** will fold in solution to adopt a conformation amendable for coupling of both bridges at the same time (Figure 45).

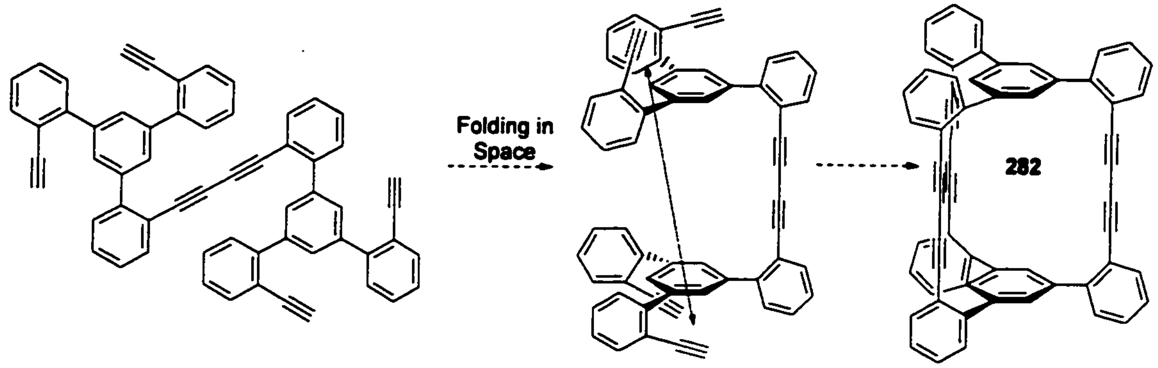


Figure 45. Required conformation of 326 to form the desired cyclophane.

Instead, assuming sequential formation of the second and third bridges of **282**, it is statistically more likely that rotation of **326** would cause one of the acetylenes to encounter the wrong coupling partner (Figure 46). The dimerization of acetylenes is fast and irreversible, locking the molecule in the conformation represented by the cyclophane, **284**. If the molecule were to spin in the opposite direction, it would also result in the formation of **284** instead of the desired **282**.

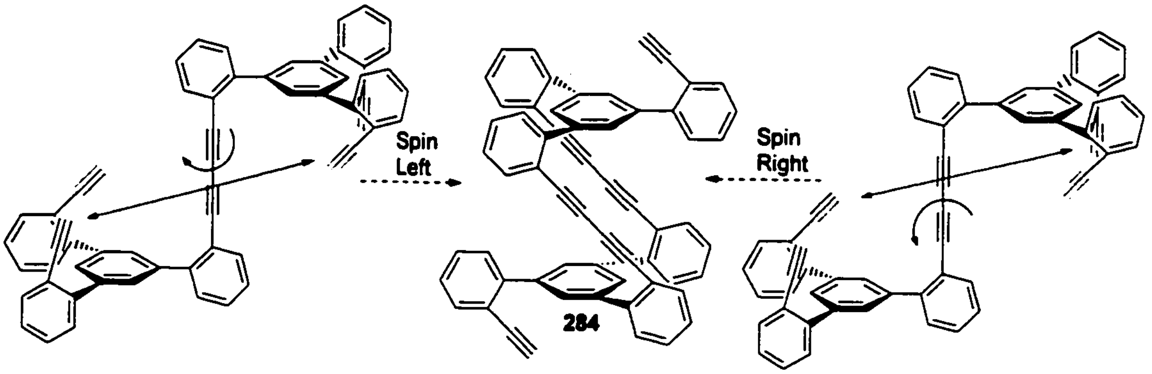


Figure 46. Folding of 326 in space statistically gives the product 284 over 282.

5.4 Progress Towards Sequential Coupling Routes to 1,3,5-Trisubstituted Cyclophanes Employing a Double Cyclization Sonogashira Reaction.

Encouraged by the results of the sequential coupling approach to **282** using a double dimerization of acetylene for the installation of the two final butadiyne bridges, a second sequential approach was investigated. The sequential coupling strategy was identical to that employed by Moore and co-workers for the construction of various 3-dimensional nanostructures.[31,32] This strategy would require the alteration of our desired target from **282** to the structure represented by **327** (Figure 47). The structure would not possess butadiyne linkages but is still composed of 60 carbon atoms. A double cyclization of **328** would result in **327** and could be obtained from the coupling of precursors **329** and **330** (Figure 47).

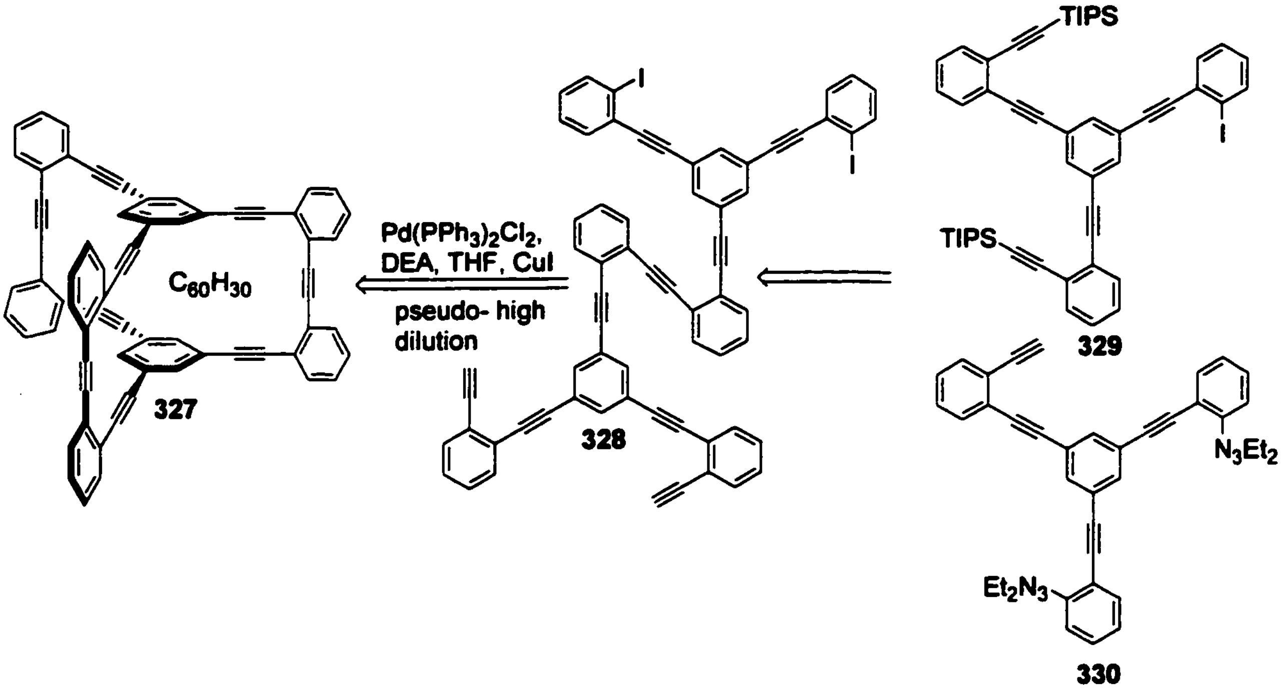
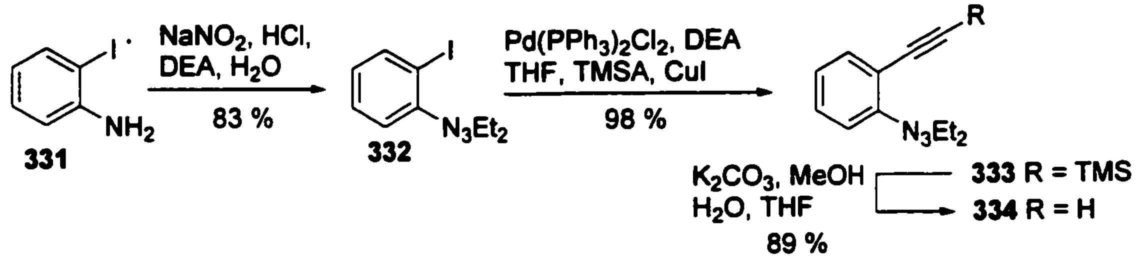


Figure 47. Sequential coupling approach to 60-carbon cyclophane employing a final intramolecular Sonogashira coupling.

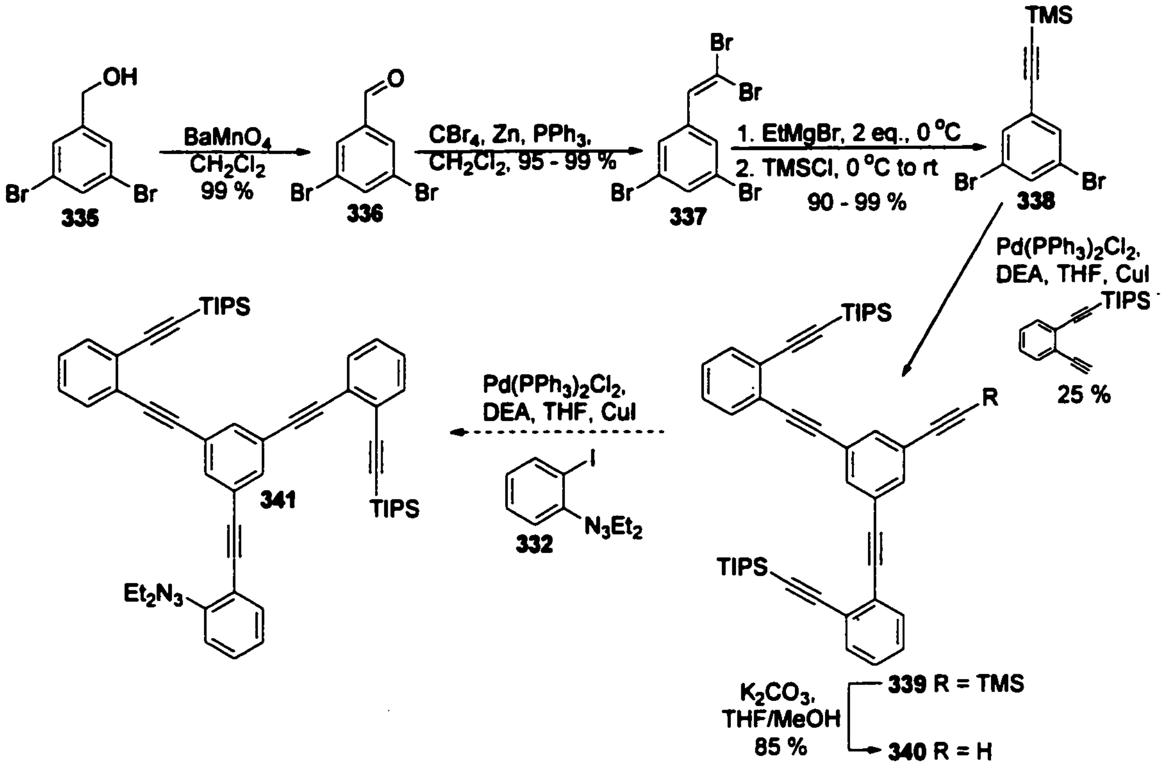
The precursors could in turn be constructed using the chemistry developed previously by Haley and co-workers (Scheme 85).



Scheme 85. Synthesis of the building blocks 332 and 334.

The construction of precursor **329** is described in Scheme 86. Oxidation of 3,5-dibromobenzyl alcohol with barium manganate in dichloromethane gave the corresponding benzaldehyde in 99% yield.[31,32,33] Corey-Fuchs reaction subsequently gave the desired dibromide, **337** in 95 – 99% yield and the bromide could be converted to the acetylene, **338**, in 90% yield, by elimination with two equivalents of ethyl magnesium bromide followed by quenching of the resulting acetylide with TMSCl.[31,32,33]

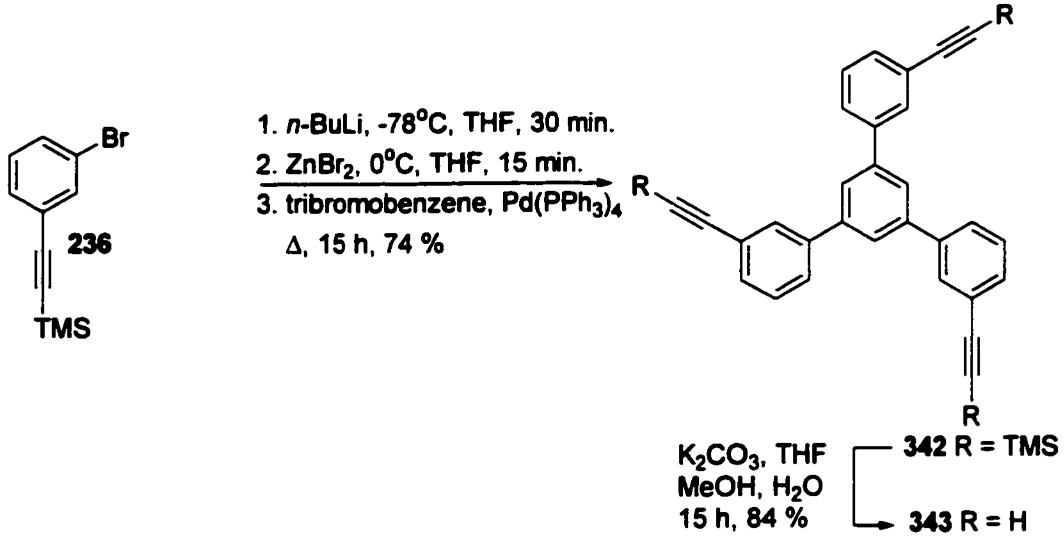
Sonogashira coupling of two equivalents of **319** with **338** occurred in poor yields to give **339** in approximately 25%. The trimethylsilyl protecting group could be removed selectively with potassium carbonate to give the terminal acetylene, **340** in 85% yield. The coupling of **332** to the terminal acetylene and removal of the triazene protecting group to the corresponding iodide by refluxing in MeI in a sealed tube remained to give the precursor, **329**.



Scheme 86. Sequential coupling approach to 60-carbon cyclophane employing a final intramolecular Sonogashira coupling.

5.5 Synthesis and Design of 1,3,5-Trisubstituted Cyclophanes Bearing *meta*-Phenyl-Yne Linkages.

The interesting results in the studies of 1,3,5-trisubstituted cyclophanes bearing *ortho*-phenyl-yne linkages suggested the design of a second family bearing *meta*-phenyl-yne bridges. With the building block, **236** in hand, generation of the organozincate and palladium-catalyzed cross-coupling to 1,3,5-tribromobenzene gave the TMS protected product, **342** in 74% yield (Scheme 87). The TMS protecting groups were removed under basic conditions with potassium carbonate in MeOH and THF to give **343** in 84% yield. The triacetylene, **343**, was then added by syringe pump under pseudo high dilution conditions to minimize polymerization of the starting material. The addition of **343** to a solution of various copper catalysts failed to produce any soluble organic products (Table 15).



Scheme 87. Synthesis of 1,3,5-trisubstituted cyclophanes bearing meta-phenyl-yne linkages.

*Table 15. Attempts at dimerizing, **343**.*

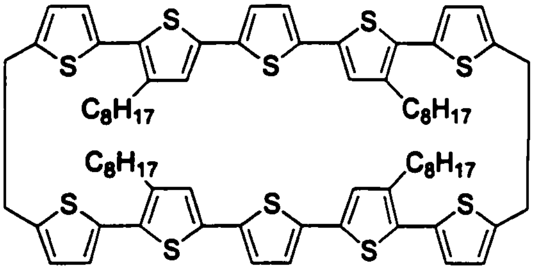
Entry	Conditions
1	CuCl, TMEDA, O_2, benzene, 15 h
2	$Cu(OAc)_2$ (100 eq.), 3:1 py: Et_2O, rt, 4 h
3	$Cu(OAc)_2$ (9 eq.), 3:1 py: Et_2O, rt, 4 h
4	$Cu(OAc)_2{\cdot}H_2O$ (9 eq.), CH_3CN, 60 °C, 4 h
5	$Cu(OAc)_2$ (25eq.), CuCl (20 eq.), py, 60 °C, 4 h

CHAPTER 6: PROGRESS TOWARDS PHENYL/ACETYLENE THIENOPHANES AND THIOPHENYL/ACETYLENE CYCLOPHANES.

6.1 Stepwise Construction of Thiophenyl/Acetylene Cyclophanes.

Polythiophenes and their finite oligomers, α-conjugated oligothiophenes, remain one of the most frequently investigated conjugates systems.[151] Their conjugated systems result in interesting redox states, outstanding electronic properties, and hence, have great potential in electronic devices and organic light emitting diodes, lasers, and transistors.

Thiophenes are easily functionalized and the chain lengths of oligothiophenes are controllable, allowing for structure-relationship studies to be pursued. Cyclic oligothiophenes are often investigated for comparison with linear models. Otsubo and co-workers have studied the [2.2]quinquethiophenophane, **344**, as an ideal π-dimer model that mirrors the behavior of oligothiophenes in the solid or solution state (Figure 48).[152]



344

Figure 48. The [2.2]quinquethiophenophane, 344 acts as a model π-dimer to investigate the electrical properties of linear oligothiophenes.

Macrocycles bearing thiophene and other conjugated units represent model systems that ideally combine the π-conjugation of an idealized infinite polymer and the well-defined structural characteristics of an oligomer. Many cyclic structures possess cavities and may form interesting inclusion complexes. Other conjugating angular units such as a double bond often connect the thiophene units of the macrocycles, but are often not as efficient at continuing conjugation as alkynes.[153] The electrical properties of

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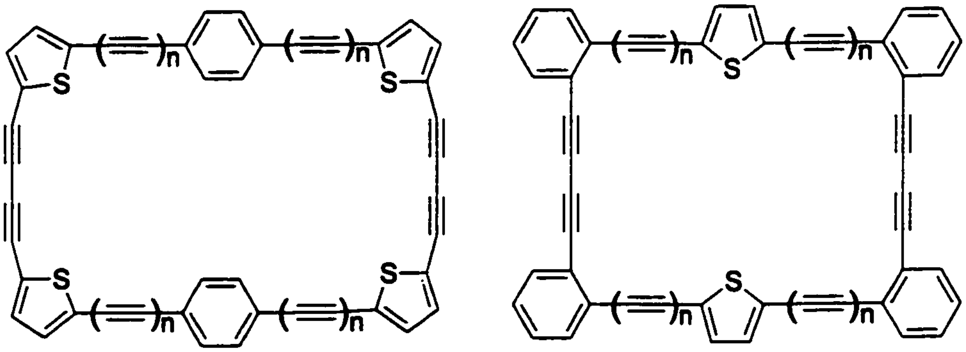
[151] (a) *Handbook of Oligo- and Polythiophenes;* Fichou, D., Ed.; Wiley-VCH, Weinheim, Germany, **1999**. (b) "Oligothiophenes", Baurele, P. in *Electronic Materials: The Oligomer Appraoch*; Mullen, K.; Wegner, G., Eds.; Wiley-VCH, Weinheim, **1998**, pp. 105-197. (c) Martin, R.E.; Deiderich, F. *Angew. Chem. Int. Ed. Engl.* **1999**, *38*, 1350. (d) Tour, J.M. *Chem. Rev.* **1996**, *96*, 537.

[152] Kaikawa, T.; Takiyama, K.; Aso, Y.; Otsubo, T. *Org. Lett*, **2000**, *2*, 4197.

[153] Bunz, U.H.F. *Top. Curr. Chem.* **1999**, *201*, 131.

connected thiophenes are maximized when conjugated at the α,α-positions, and are not as efficient for the corresponding α,β or β,β isomers.[154]

Investigations into the incorporation of thiophenes into phenyl/acetylene macrocycles were commenced (Figure 49). The thiophenes would be connected at the α-positions and the macrocycles could be examined for interesting electrical properties. These molecules would be adaptations of our previously studied ene-yne and phenyl-yne cyclophanes. The number of acetylene linkages could be altered and the thiophene could either occupy the angular units in the cyclophane (left, Figure 49) or the central position (right, Figure 49).



345 n = 0
346 n = 1

347 n = 0
348 n = 1

Figure 49. Basic design of phenyl/acetylene thiophenophanes.

Similar types of cyclophanes have already been synthesized by Marsella and co-workers.[97] They constructed cyclophanes where the thiophene units were connected at the α- and β-positions by a double Sonogashira coupling outlined in Scheme 88. In their continuing studies on the construction of molecules bearing helical type architectures, they examined the effect of the differing quadrupole moments of perfluorophenyl and phenyl groups on cyclophane cyclization. The interactions or attraction of the two phenyl moieties are responsible for cyclization as opposed to random polymerization of intermediates. Thus higher yields of the cyclophane **354** were observed in comparison to cyclophanes **353** and **355**.

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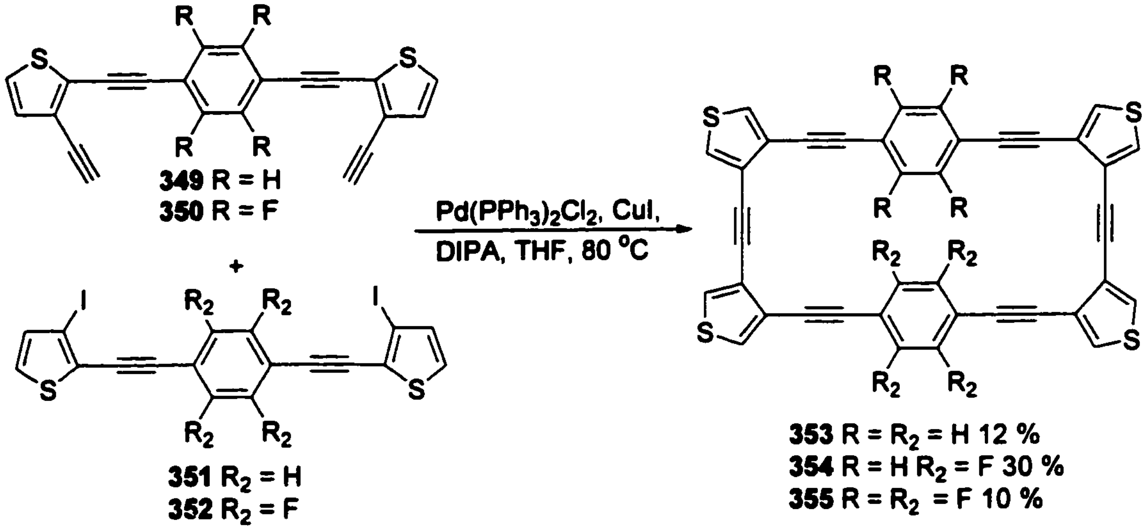
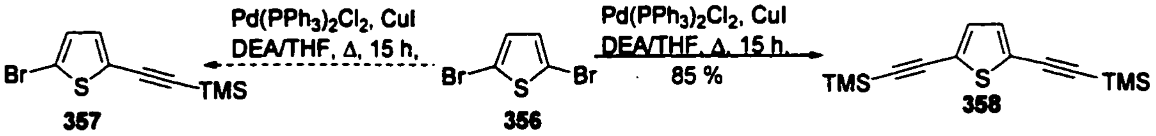
[154] Kauffman, T. *Angew. Chem. Int. Ed. Engl.* **1979**, *18*, 1.



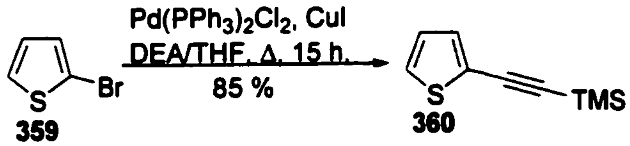
Scheme 88. Marsella's cyclization of thiophenic cyclophanes was improved using perfluorophenyl-phenyl interactions to preorganize starting materials.

For the purpose of constructing cyclophanes **345** and **346**, the building block, 2-bromo-5-(trimethylsilylethynyl)thiophene, **357**, was required. Precursor, **357** would be analogous to the phenyl building blocks **185** and **186**. Monocoupling of TMSA with dibromo-precursor, **356** by a Sonogashira reaction was not possible as the dicoupling product, **358**, was isolated as the only product (Scheme 89).



Scheme 89. Failed synthesis of 357.

Alternatively, compound **360** was synthesized by palladium catalyzed cross-coupling of TMSA to 2-bromothiophene (Scheme 90). The proton at the 5-position can be deprotonated with lithium bases for the synthesis of 5-substituted thiophenes.[155]



Scheme 90. Construction of the building block, 360.

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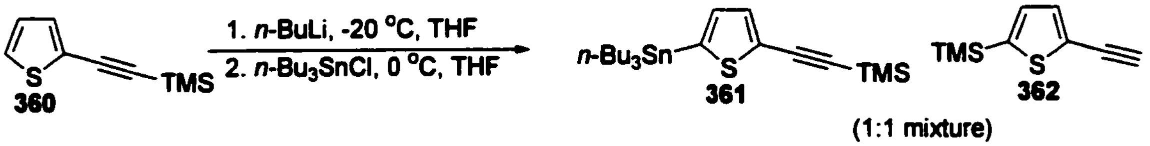
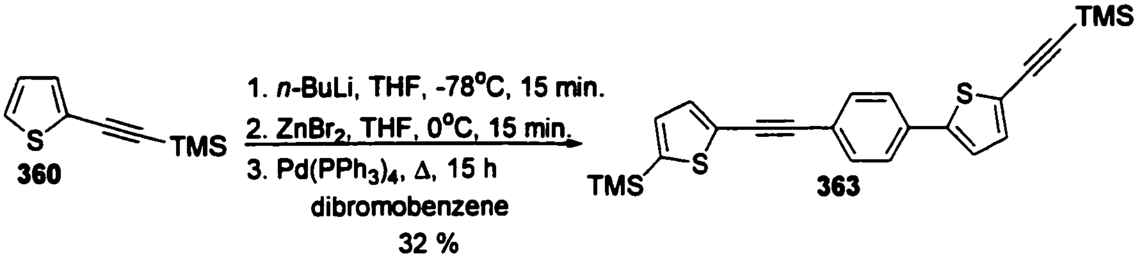
[155] (a) Chadwick, D.J.; Willbe, C. *J. Chem. Soc. Perkin Trans. I* **1976**, 887. (b) Feringa, B.L.; Hulst, R.; Rikrs, R.; Brandsma, L. *Chem. Commun.* **1987**, 316. (c) Furukawa, N.; Hoshiai, H.; Shibutani, T.; Higaki, M.; Iwasaki, F.; Fujihara, H. *Heterocycles* **1992**, *34*, 1085.

Unfortunately, treatment of **360** with *n*-BuLi at –20 °C and quenching with tri-*n*-butylstannylchloride caused a 1:1 mixture of two products. ^{1}H NMR analysis of the products identified them as **361** and **362** (Scheme 91). The products originate from a Brook-like rearrangement as the lithium salt of **360** may remove a TMS group from another molecule or may be competitively quenched by the trialkylstannyl group.



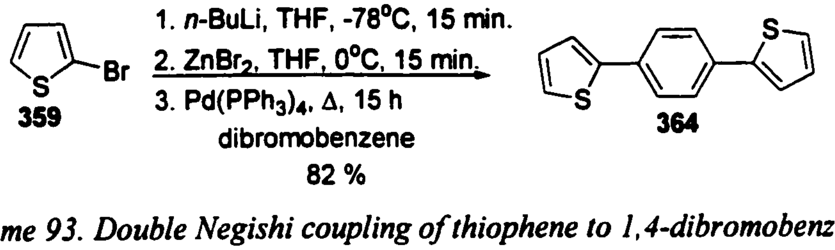
Scheme 91. Rearrangement of the trimethylsilyl group of 360 under basic conditions.

The quenching may be slow due to the large steric bulk of the tri-*n*-butylstannyl group. Thus transmetallation with $ZnBr_2$ with conducted instead and the organozincate was cross-coupled with 1,4-dibromobenzene (Scheme 92). A Negishi coupling gave a single product that was identified by ^{1}H NMR as **363**. The product would result from coupling of a mixture of products resulting from transmetallation products of the ZnBr being attached either to a terminal acetylene or to the desired α-position of a thiophene.



Scheme 92. Migration of the TMS group results in a mixed Negishi coupling product.

Palladium catalyzed cross-coupling of thiophenes are highly efficient, thus a Negishi coupling was investigated.[156] 2-Bromothiophene, **359**, could undergo lithium-halogen exchange, transmetallation with $ZnBr_2$, and a palladium mediated cross-coupling to give **364** in 82% yield.



Scheme 93. Double Negishi coupling of thiophene to 1,4-dibromobenzene.

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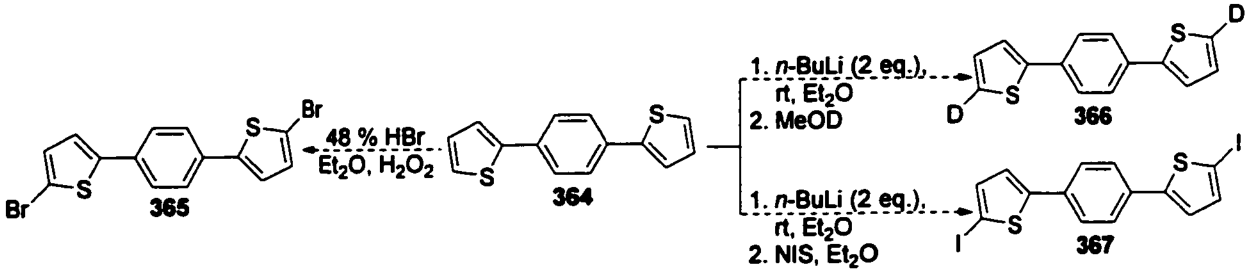
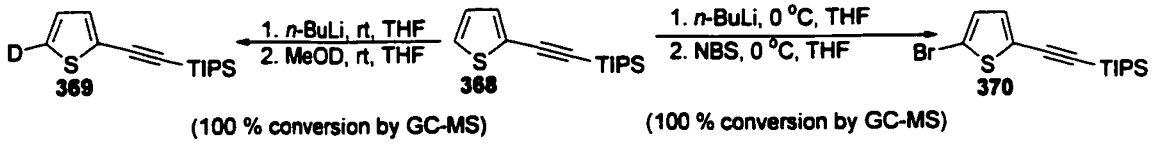
[156] (a) Ohta, A.; Akita, Y.; Ohkuwa, T.; Chiba, M.; Fukunaga, R.; Miyafuji, A.; Nakata, T.; Tani, N.; Aoyagi, Y. *Heterocycles* **1990**, *31*, 1951. (b) Bailey, T.R. *Tetrahedron Lett.* **1986**, *37*, 4407.

Unfortunately, functionalization at the α-positions was difficult. Double deprotonation with *n*-BuLi was not possible as quenching with deuterium or iodide gave mixtures of different products (Scheme 94). Similarly, oxidative bromination of the α-position of the thiophene rings in **364** using hydrogen peroxide and 48% HBr (aq) also caused the formation of a complex mixture of products (Scheme 94).



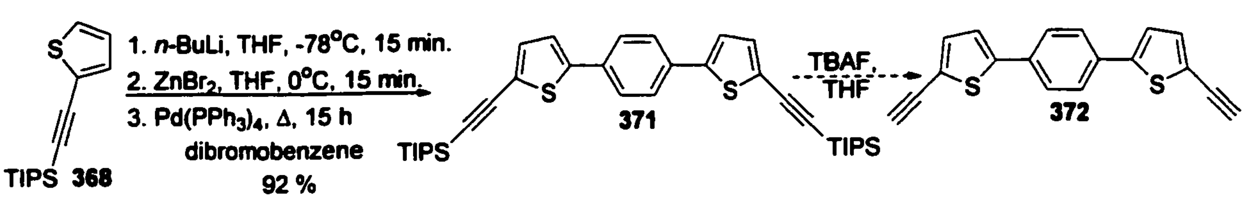
Scheme 94. Functionalization of the α-positions of 364.

Consequently, to inhibit migration of the trimethylsilyl group, **368** was synthesized by an analogous Sonogashira coupling. The installation of a large and bulky triisopropylsilyl group should stop any migration. Treatment of **368** with *n*-BuLi at 0 or –20 °C showed no migration of the silyl group by GC-MS analysis. Quenching with *d*-MeOH or NBS gave 100% conversion to the corresponding deuterated, **369** and the bromide, **370** (Scheme 95).



Scheme 95. Quenching of the lithium salt of 368 gave the corresponding deuterated and brominated compounds, 369 and 370, without migration of the trialkylsilyl group.

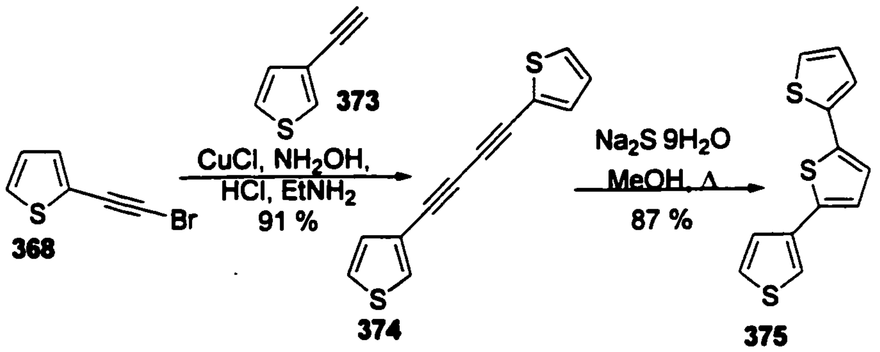
Thiophene **368** could be successfully deprotonated and transmetallated to undergo Negishi coupling with 1,4-dibromobenzene to give **371** as a bright fluorescent yellow powder in 92% yield (Scheme 96). The subsequent desilylation resulted in the solution turning black upon addition of TBAF and surprisingly all starting material was consumed.



Scheme 96. Synthesis of thiophene precursors via *Negishi coupling.*

6.2 Incorporation of Thiophenes Into Phenyl/Acetylene Cyclophanes.

Well-defined oligothiophenes can be constructed by palladium catalyzed cross-coupling of thiophenes, but α,α-connected thiophenes can also be installed by treating butadiyne units with sodium sulfide in refluxing methanol.



Scheme 97. Kagan's synthesis of terthiophenes.

Kagan and co-workers studied the formation of different substituted terthiophenes and examined their photosensitizing abilities.[157] Oligothiophene, **375** was previously synthesized using Grignard chemistry but could be assembled by treatment of butadiyne, **374**, with sodium sulfide in alcohol.

Bäurele and co-workers have constructed large α-conjugated macrocyclic oligothiophenes using a similar strategy (Scheme 98). Their construction of large cyclic[*n*]thiophenes with tunable cavities is based on a dimerization of acetylenes strategy to form cyclic thienophanes bearing butadiyne linkages . These linkages are reacted with sodium sulfide in a solvent mixture of 2-methoxyethanol and xylenes to produce the large macrocycle, **378**. These molecules self assemble in the solid state with cavities in the range of 2-3 nm. Bäurele and co-workers are currently investigating the inclusion complexes with C_{60} and the possibility of forming novel nanotubes.

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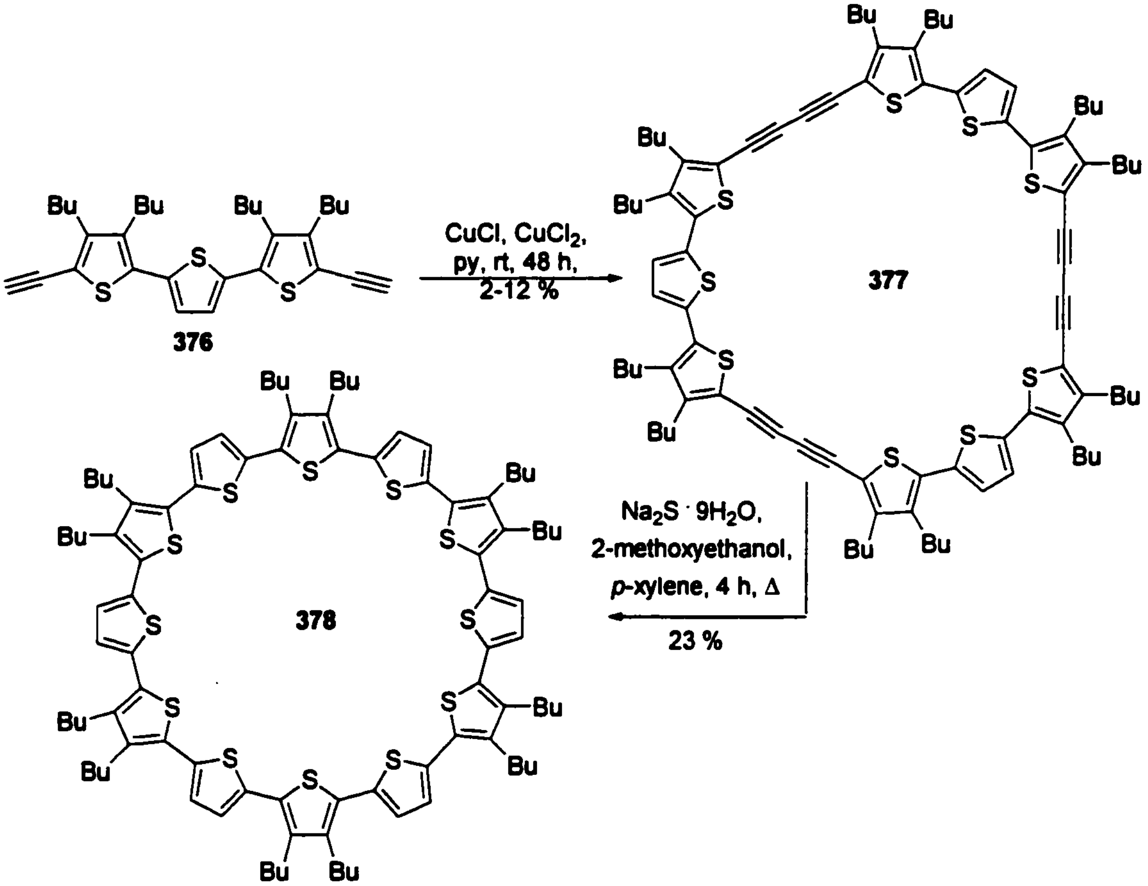
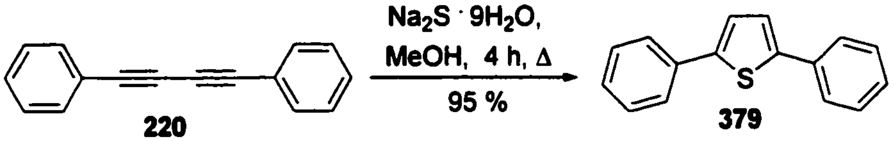
[157] Kagan, J.; Arora, S.K.; Prakash, I.; Ustunol, A. *Heterocycles* **1983**, *20*, 1341.



Scheme 98. Construction of large cyclic[n]thiophenes.

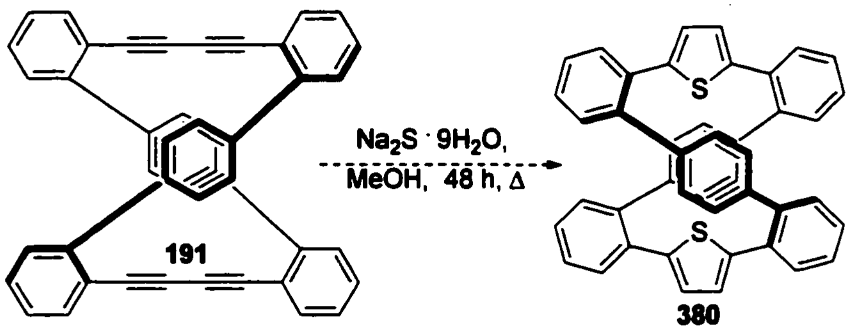
It was established by Kagan[157] and this laboratory that 1,4-diphenylbutadiyne undergoes smooth conversion to 2,5-diphenylthiophene in 95% yield when treated with sodium sulfide in refluxing methanol (Scheme 99).



Scheme 99. 1,4-diphenylbutadiyne undergoes smooth conversion to the corresponding 1,4-diphenylthiophene.

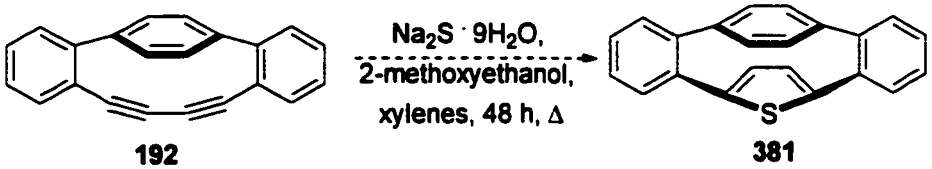
Consequently, the 1,4-diphenylbutadiyne linkages present in the dimer, **191** were subjected to sodium sulfide to form α,α-connected thiophenes and generate the helical macrocycle, **380** (Scheme 100). Dimer **191** was not soluble in methanol and reacting the molecule as a suspension produced only recovered starting material. Use of benzene or benzene/methanol/ethanol solvent mixtures were also incapable of solubilizing the dimer.

Using the 2-methoxyethanol/xylenes solvent system solubilized all reactants but no reaction was observed.



*Scheme 100. Failed conversion of **191** to the thiophenic cyclophane, **380**.*

Cyclophane **192**, possessing the strained butadiyne linkage, was dissolved in 2-methoxyethanol/xylenes and treated with sodium sulfide. However, before the reaction temperature reached reflux, the reaction mixture had turned a deep black colour and a mixture of products had formed with complete consumption of the starting material. None of the isolated products resembled the desired macrocycle, **381**. Currently, the addition of crown ethers and replacement of the sodium cation with potassium are being investigated to increase the reactivity of the sulfide anion.



*Scheme 101. Attempted synthesis of **381**.*

CLAIMS TO ORIGINAL RESEARCH.

1. Synthesized a family of novel acetylenic [1.4]cyclophanes *via* a dimerization strategy and demonstrated that the termini separation between acetylenes is a contributing factor in competing intramolecular cyclizations.
2. Elucidation of the solid state X-ray crystal structures of cyclophanes, **191** and **213** confirmed that these structures adopt twisted helical geometries.
3. Developed and optimized efficient methods for the *in situ* desilylation and dimerization of acetylenes to form diynes and tetraynes.
4. Constructed a series of acetylenic cyclophanes bearing long aliphatic chains with potential as novel liquid crystals.
5. Synthesized several highly strained acetylenic cyclophanes. X-ray crystal analysis revealed that **307** possessed one of the highest recorded deformations of carbon-carbon triple bonds for butadiynes. The alkenic nature of the strained bonds was established through Diels-Alder cycloadditions.
6. Established sequential coupling protocols as an alternative synthetic approach to cage acetylenic structures when traditional methods are hampered by intramolecular cyclization.
7. Developed initial approaches to various C_{60} caged systems whose refinements should lead to novel cyclophanes and perhaps fullerene precursors.

Publications

1. Collins, S.K.; Yap, G.P.A.; Fallis, A.G. *Angew. Chem. Int. Ed.* **2000,** *39*, 385.
2. Heuft, M.A.; Collins, S.K.; Yap, G.P.A.; Fallis, A.G. *Org. Lett.* **2001,** *3*, 2883.
3. Collins S.K., Yap G.P.A., Fallis, A.G. *Org. Lett.* **2000,** *20*, 3189

General. Proton magnetic resonance and carbon magnetic resonance spectra (^{1}H NMR) were measured at 500 MHz with a Bruker AMX500 or at 200 MHz with a Varian Gemini spectrometer. Chemical shifts are reported in parts per million (ppm) downfield from tetramethylsilane (δ scale). The multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad), number of protons and coupling constants (reported in Hz) are indicated in parentheses. Electron impact mass spectra EI (MS) were determined on a Kratos Concept 2H instrument using an ionization energy of 70 eV. Electrospray mass spectra ES (MS) were determined on a Micromass Quattro LC with a pump rate of 20 μL/min. Elemental analyses were performed at M-H-W Laboratories, Phoenix, Arizona, USA. The purity of all title compounds was judged to be > 95% as determined by a combination of GC-MS, ^{1}H NMR and ^{13}C NMR analyses.

All non-aqueous reactions were performed under an atmosphere of dry nitrogen or dry argon in flame or oven dried glassware equipped with a magnetic stir bar and a rubber septum. Room temperature corresponds to 23 °C. Standard inert atmosphere techniques were used in handling all air and moisture sensitive reagents. Reactions were monitored by analytical thin layer chromatography (TLC) using commercial aluminum sheets pre-coated (0.2 mm layer thickness) with silica gel 60 F_{254} (E. Merck). Organic layers after separations were dried over anhydrous magnesium sulfate unless otherwise specified. Product purification by chromatography refers to conventional and flash column chromatography performed using E. Merck Silica Gel (70-230 or 230-400 mesh). Petroleum ether refers to a mixture of hydrocarbons with a boiling range of 30 – 60 °C. Anhydrous diethyl ether (ether), anhydrous tetrahydrofuran (THF) were freshly distilled from benzophenone/sodium. Dry benzene, toluene, dimethylformamide (DMF), dichloromethane, and triethylamine were distilled from NaH or CaH. $Cu(OAc)_2$ was prepared from the dihydrate $Cu(OAc)_2 \cdot 2H_2O$ by refluxing in acetic anhydride for 15 h prior to use.[158] *N*-BuLi was used as commercially available solutions in hexanes from Aldrich Chemical Company and titrated prior to use against diphenylacetic acid. All TBAF solutions were in THF solvent. All commercial starting materials were purchase from Aldrich Chemical Company unless otherwise stated.

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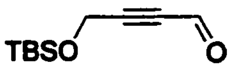
[158] Spath, E. *Sitzungsber. Akad. Wiss. Wien. Math.-Naturwiss. Kl., Abt. 2B* **1911**, *120*, 117.

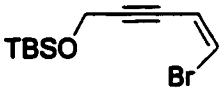
4-(tert-Butyl-dimethyl-silanyloxy)-but-2-ynal



157

Compound **156** (200 mg, 1.0 mmol), and Dess-Martin Periodinane (618 mg, 1.50 mmol) were dissolved in dichloromethane (10 mL) and cooled to 0 °C. $NaHCO_3$ (205 mg) was added and the reaction stirred for 10 minutes. The reaction mixture was diluted with 1:1 ($Na_2S_2O_3$ (sat): H_2O) (10 mL) and stirred until reaction mixture was clear. The reaction mixture was separated between diethyl ether and water. The organic phase was dried, filtered and evaporated to yield the product as a clear oil (168 mg, 85%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.21 (d, *J* = 0.4 Hz, 1H), 4.48 (s, 2H), 0.89 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.4 (d), 94.9 (s), 84.2 (s), 51.6 (t), 25.8 (q), -5.26 (q); IR (neat): *v* = 2955, 2857, 2333, 1701, 1584, 1463, 1255, 1082, 836 cm^{-1}; MS (EI) *m/z* 198 (M^+), 183, 141, 127, 111, 99, 83, 75, 57, 45.

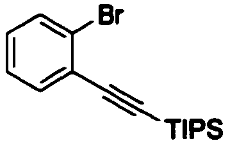
(5-Bromo-pent-4-en-2-ynyloxy)-tert-butyl-dimethyl-silane



159

1-Bromomethyltriphenylphosphonium bromide (203 mg, 0.46 mmol) was dissolved in THF (10 mL) and cooled to –78 °C. Potassium *t*-butoxide (57 mg, 0.46 mmol) was added and the reaction mixture stirred for 30 minutes. The aldehyde **157** (84 mg, 0.42 mmol) was added as a solution in THF (5 mL) and stirred at 0 °C for 2 h. The reaction mixture was quenched with saturated ammonium chloride solution and the partitioned between diethyl ether and water. The organic phase was dried, filtered and the crude product purified by chromatography (Pet Ether to yield the product as a clear oil (32 mg, 27%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.51 (d, *J* = 7.7 Hz, 1H), 6.29 (d, *J* = 7.6 Hz, 1H), 4.42 (d, *J* = 1.7 Hz, 2H), 0.88 (s, 9H), 0.10 (s, 6H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 117.6 (d), 115.3 (d), 95.9 (s), 80.4 (s), 52.2 (t), 25.8 (q), -5.13 (q); IR (neat): *v* = 2955, 2857, 2333, 1584, 1463, 1255, 1082, 836 cm^{-1}; MS (EI) *m/z* 217 (M^+-*t*-Bu), 189, 163, 139, 123, 107, 83, 75, 63, 45, 29; HRMS Calcd for $C_7H_{10}OBrSi$ 216.9684, found 216.9850.

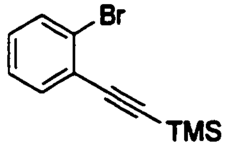
1-Bromo-2-(triispropylsilylethynyl)benzene



185

CuI (316 mg, 1.65 mmol), $Pd(PPh_3)_4$ (349.2 mg, 0.5 mmol) and 1-bromo-2-iodobenzene (7.27 g, 25.7 mmol) were added to a flame dried round bottom flask and dissolved in DEA (100 mL) and THF (100 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar or N_2. Triisopropylsilylacetylene (7.0 mL, 30.8 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide a slightly pale yellow liquid. The liquid was further purified by preparative HPLC to provide the product as a pale yellow liquid (7.78 g, 89%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (dd, J = 7.7, 1.7 Hz, 1H), 7.50 (dd, J = 8.0, 1.7 Hz, 1H), 7.22 (dt, J = 7.5, 1.2 Hz, 1H), 7.22 (dt, J = 7.8, 1.7 Hz, 1H), 1.17 (s, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) 133.8 (d), 132.3 (d), 129.3 (d), 126.8 (d), 125.7 (s), 125.6 (s), 104.8 (s), 96.1 (s), 18.6 (q), 11.3 (d); IR (neat): ν = 3064, 2943, 2865, 2161, 1558, 1464, 1433, 1220, 1027, 995, 882, 834, 753, 674 cm^{-1}; MS (EI) *m/z* 338, 336 (M^+), 296, 295, 293, 265, 253, 239, 223, 209, 207, 162, 143, 129; HRMS Calcd for $C_{17}H_{25}BrSi$ 336.2840 (M^+), found 336.0889; Anal Calcd for $C_{17}H_{25}BrSi$: C, 60.51; H, 7.48. Found: C, 60.40; H, 7.38.

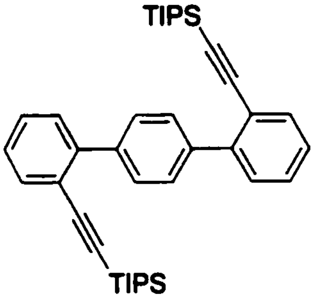
1-Bromo-2-(trimethylsilylethynyl)benzene



186

CuI (315 mg, 1.65 mmol), $Pd(PPh_3)_2Cl_2$ (355 mg, 0.51 mmol) and bromoiodobenzene (7.27 g, 25.7 mmol) were dissolved in DEA (100 mL). The resulting dark green solution was degassed for 15 min. by sparging with Ar. Trimethylsilylacetylene (3.0 g, 30.6 mmol) was added followed by THF (100 mL) to help dissolve the thick mixture. The dark green solution was brought to reflux and heated overnight under Ar. Solvent was then removed under reduced pressure and the resulting black solid was purified by chromatography (Pet. Ether) to provide the product as a slightly pale yellow liquid (5.94 g, 91%). ^{1}H NMR (200 MHz, $CDCl_3$) 7.57 (dd, J = 7.6, 1.9 Hz, 1H), 7.53 (dd, J = 7.0, 1.1 Hz, 1H), 7.23 (dt, J = 7.5, 1.5 Hz, 1H), 7.13 (dt, J = 7.0, 1.0 Hz, 1H), 0.27 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) 133.5 (d), 132.3 (d), 129.5 (d), 126.8 (d), 125.7 (s), 125.2 (s), 103.0 (s), 99.5 (s), -0.2 (q); IR (neat): ν = 3065, 2960, 2899, 2351, 2163, 1585, 1558, 1465, 1429, 1251, 1220, 1120, 1046, 1027, 945, 858, 757 cm^{-1}; MS (EI) *m/z* 254, 252 (M^+), 241, 240, 239, 238, 237, 143, 131, 115, 79, 69, 43; HRMS calcd for $C_{11}H_{13}BrSi$ 251.9970, found 251.9916.

1,4-(*o*-Triispropylsilylethynylphenyl)benzene

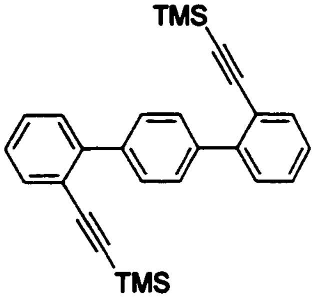


188

1-Bromo-2-(triispropylsilylethynyl)benzene, **185** (339.5 mg, 1.00 mmol) in THF (15 mL) was cooled to -78°C and *n*-BuLi (475 μL, 1.00 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (227.4 mg, 1.01 mmol) in THF (15 mL) at -78°C was transferred *via* canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (35.6 mg, 0.035 mmol) and dibromobenzene (94.1 mg, 0.39 mmol) was then added to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and partitioned between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a pale yellow solid (158.2 mg, 80%). m.p. 74-

78°C; ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.61 (m, 2H), 7.59 (s, 4H), 7.36 (m, 4H), 7.27 (m, 2H), 1.00 (s, 42H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 144.2 (s), 139.5 (s), 133.8 (d), 129.3 (d), 128.9 (d), 128.5 (d), 126.8 (d), 122.0 (s), 106.4 (s), 94.1 (s), 18.6 (q), 11.3 (d); IR (CHCl$_3$): $v = 2945, 2865, 2154, 1468.38, 995, 882, 829, 782, 756, 670$ cm^{-1}; MS (EI) *m/z* 591, 590 (M^+), 357, 319, 305, 252, 158, 157, 149, 115, 59; HRMS Calcd for $C_{40}H_{54}Si_2$ 590.8302, found 590.3759; Anal Calcd for $C_{40}H_{54}Si_2$: C, 81.27; H, 9.23. Found: C, 81.43; H, 9.49.

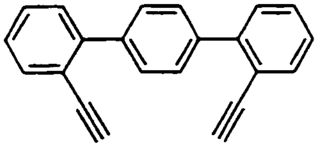
1,4-(*o*-Trimethylsilylethynylphenyl)benzene



189

1-Bromo-2-(trimethylsilylethynyl)benzene **186** (4.04 g, 15.9 mmol) in THF (150 mL) was cooled to -78°C and *n*-BuLi (6.3 mL, 2.5 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of ZnBr$_2$ (3.58 g, 16 mmol) in THF (150 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of Pd(PPh$_3$)$_4$ (450 mg, 0.39 mmol) and dibromobenzene (1.25 g, 5.29 mmol) in THF (100 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a yellow semi-solid. Alternatively, the reaction could be quenched by the addition of silica gel. Evaporating to dryness gives a crude yellow powder. Either crude product can be further purified by chromatography to give the product as a pale yellow solid (1.55 g, 70%). m.p. 166-167°C; ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.64 (s, 4H), 7.59 (d, $J = 7.5$ Hz, 4H), 7.36 (m, 2H), 7.27 (m, 2H), 0.12 (s, 18H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 144.0 (s), 139.3 (s), 133.3 (d), 129.3 (d), 128.7 (d), 126.8 (d), 121.4 (s), 104.8 (s), 97.7 (s), -0.31 (q); IR (CHCl3): v = 3059, 3015, 2959, 2896, 2151, 1469, 1431, 1250, 1212 cm^{-1}; MS (EI) *m/z* 422 (M$^+$), 319.1, 289, 252, 215, 162, 112, 73, 45; HRMS Calcd for $C_{28}H_{30}Si_2$ 422.1887, found 422.1869.

1,4-(*o*-Ethynylphenyl)benzene

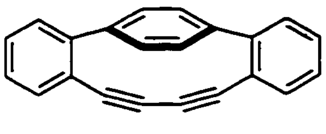


190

From **188**: 1,4-(*o*-Triispropylsilylethynylphenyl)benzene, **188** (130.2 mg, 0.22 mmol) was dissolved in THF (7 mL) and TBAF (440 μL, 1.0 M) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was further purified by chromatography to give the product as a yellow solid (64 mg, 100%).

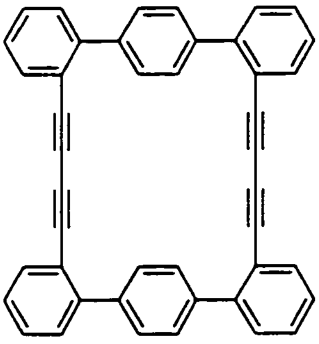
From **189**: 1,4-(*o*-Trimethylsilylethynylphenyl)benzene, **189** (1.5 g, 3.6 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a white solid (920 mg, 90%). m.p. 195-196°C; ^{1}H NMR (500 MHz, CDCl3) δ 7.66 (s, 4H), 7.62 (dd, J = 7.8, 1.0 Hz, 2H), 7.43 (m, 4H), 7.30 (dt, J = 7.5, 1.7 Hz, 2H), 3.06 (s, 2H); ^{13}C NMR (125 MHz, CDCl3) δ 143.9 (s), 139.3 (s), 134.0 (d), 129.6 (d), 129.0 (d), 128.8 (d), 127.0 (d), 120.4 (s), 83.1 (s), 80.2 (d); IR ($CHCl_3$): v = 3309, 2101, 1463, 1219 cm^{-1}; MS (EI) *m/z* 278 (M$^+$), 252, 217, 138, 85, 71, 57; HRMS Calcd for $C_{22}H_{14}$ 278.1096, found 278.1075; Anal Calcd for $C_{22}H_{14}$: C, 94.92; H, 5.08. Found: C, 94.96; H, 5.20.

1,3-Diyne-[4]-(2,2'')-[1,1';4',1'']terphenylophane



192

1,3,13,15-Tetrayne-[4.4]-(2,2")-[1,1';4',1"]terphenylophane



191

$Cu(OAc)_2$ (250 mg, 6 equiv.) was dissolved in pyridine: ether (3:1) (70 mL) and 1,4-(*o*-ethynylphenyl)benzene (64.2 mg, 0.23 mmol) in 20 mL of pyridine:ether (3:1) was added over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel could be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids could be further purified by chromatography (6:1 Hexanes: CH_2Cl_2) to yield the monomer product **192** as a yellow solid (38 mg, 52%) and the dimer product **191** as a pale yellow solid (19 mg, 26%).

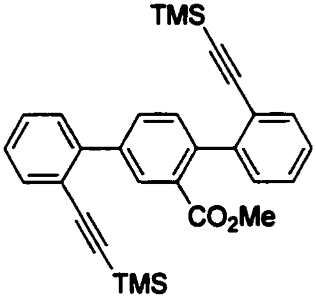
192 m.p.= 167-170 °C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.72 (m, 3H), 7.39 (m, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 150.9 (s), 141.5 (s), 129.8 (d), 128.7 (d), 127.4 (d), 126.9 (d), 126.4 (d), 124.8 (s), 98.1 (s), 84.1 (s); IR ($CHCl_3$): ν = 2965, 2921, 1508, 1457, 1260, 1099, 1019 cm^{-1}; MS (EI) *m/z* 276 (M^+), 248, 198, 162, 137, 99, 70, 31; HRMS Calcd for $C_{22}H_{12}$ 276.0940, found 276.0938.

191 m.p.= 257-260°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.4 Hz, 4H), 7.53 (s, 8H), 7.36 (m, 8H), 7.27 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2 (s), 139.3 (s), 133.7 (d), 129.5 (d), 129.2 (d), 128.8 (d), 127.1 (d), 120.4 (s), 81.3 (s), 76.7 (s); IR

($CHCl_3$): ν = 3055, 3029, 2924, 2854, 2216, 1468, 1439, 1220, 840 cm^{-1}; MS (FAB) *m/z* 551 (M^++H), 461, 391, 369, 338, 277, 246, 219.

Crystal size 0.30 x 0.04 x 0.04 mm^3, orthorhombic, space group *Pbca*, scan range 3.96<2θ<41.98°, a=15.201(5), b=20.611(6), c=21.317(6) Å, ß=90°, *V*=6679(3) Å^3, Z=8, ρ_{calcd}=1.268 g cm-3, u=0.226 mm^{-1}, 3586 unique reflections at -80°C, of which 3586 were taken as observed [I_O>2.00σ(I)], R=0.1125, R_W=0.2685. Crystallographic data (excluding structure factors) for this structure has been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-133375. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

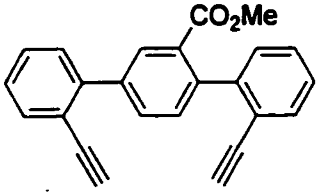
2,2''-Bis-trimethylsilanylethynyl-[1,1';4',1'']terphenyl-2'-carboxylic acid methyl ester



200

1-Bromo-2-(trimethylsilylethynyl)benzene, **186** (520 mg, 2.04 mmol) in THF (20 mL) was cooled to -78°C and *n*-BuLi (863 μL, 2.04 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (461 mg, 2.04 mmol) in THF (10mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (70 mg, 0.060 mmol) and **197** (200 mg, 0.68 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a pale yellow solid (272 mg, 80%). m.p. 113-116°C; ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.20 (d, *J* = 1.8 Hz, 1H), 7.83 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.58 (m, 1H), 7.48 (m, 1H), 7.40 (m, 3H), 7.30 (m, 4H), 3.66 (s, 3H), 0.11 (s, 9H), 0.02 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 167.5 (s), 145.0 (s), 143.0 (s), 141.0 (s), 139.6 (s), 133.4 (d), 132.4 (d), 131.6 (d), 130.8 (d), 130.7 (d), 130.3 (s), 129.3 (d), 128.9 (d), 128.4 (d), 128.4 (d), 127.3 (d), 126.9 (d), 122.3 (s), 121.5 (s), 104.4 (s), 103.8 (s), 98.1 (s), 97.6 (s), 51.9 (q), -0.32 (q), -0.40 (q); IR (CHCl$_3$) *v* = 3006, 2957, 2156, 1722, 1474, 1437, 1312, 1248, 1208, 1118 cm^{-1}; MS (EI) *m/z* 480 (M$^+$), 465, 435, 407, 391, 376, 348, 333, 319, 303, 289, 263, 225, 201, 147, 89, 73, 59; HRMS calcd for $C_{30}H_{34}O_2Si_2$, 480.1942, found 480.1927; Anal Calcd for $C_{30}H_{34}O_2Si_2$: C, 74.95; H, 6.71. Found: C, 75.05; H, 6.65.

2,2''-Bis-ethynyl-[1,1';4',1'']terphenyl-2'-carboxylic acid methyl ester

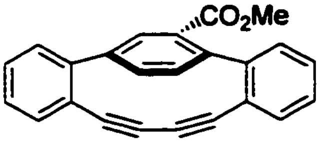


201

Compound **200** (249 mg, 0.52 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography gave the product as a fluffy yellow solid (173 mg, 99%); ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.18 (d, *J* = 1.9 Hz, 1H), 7.81 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.63 (m, 1H), 7.55 (m, 1H), 7.42 (m, 4H), 7.32 (m, 3H), 3.65 (s, 3H), 3.07, (s, 1H), 2.92 (s, 1H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 167.8 (s), 145.0 (s), 142.9 (s), 140.6 (s), 139.6 (s), 134.0 (d), 132.5 (d), 132.3 (d), 131.0 (d), 130.9 (d), 130.4 (s), 129.5 (d), 129.1 (d), 128.9 (d), 128.6 (d), 127.5 (d), 127.1 (d), 121.1 (s), 120.5 (s), 82.7 (s), 82.3 (s), 80.7 (d), 80.2 (d), 52.0 (q);

IR ($CHCl_3$) v = 3305, 1721, 1218, 1013 cm^{-1}; MS (EI) *m/z* 336 (M^+), 305, 276, 224, 200, 162, 131, 99, 59; HRMS Calcd for $C_{24}H_{16}O_2$, 336.1200, found 336.1171.

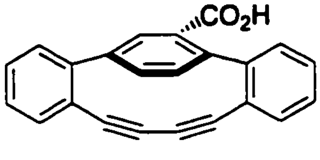
1,3-Diyne-[4](2,2")[1,1';4',1"]-2'-carboxymethylterphenylophane



202

$Cu(OAc)_2$ (483 mg, 6 equiv.) was dissolved in refluxing pyridine: ether (3:1) (150 mL) and **201** (150 mg, 0.44 mmol) in 20 mL of pyridine:ether (3:1) was added over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was cooled and poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and was washed with brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel could be added to the cooled reaction mixture and the solvent removed under reduced pressure. The crude solids could be further purified by chromatography (5% Ethyl Acetate: Pet Ether) to yield the product as a fluffy yellow solid (113 mg, 76%); m.p. 143-146°C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 1.7 Hz, 1H), 7.73 (d, *J* = 7.1 Hz, 1H), 7.69 (d, *J* = 6.8 Hz, 1H), 7.45 (m, 3H), 7.33 (m, 3H), 7.24 (m, 2H), 3.66 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4 (s), 150.5 (s), 149.4 (s), 142.7 (s), 142.1 (s), 133.4 (d), 132.3 (d), 132.0 (d), 130.9 (s), 128.8 (d), 128.6 (d), 127.8 (d), 127.2 (d), 127.0 (d), 127.0 (d), 126.9 (d), 126.4 (d), 124.9 (s), 123.1 (s), 98.0 (s), 97.9 (s), 84.8 (s), 83.3 (s), 51.8 (q); IR ($CHCl_3$) v = 2999, 2954, 2858, 2169, 1720, 1436, 1308, 1213, 1140 cm^{-1}; MS (EI) *m/z* 334 (M^+), 303, 274, 143, 112, 69, 40; HRMS calcd for $C_{24}H_{14}O_2$, 334.0094, found 334.1018.

1,3-Diyne-[4]-(2,2")-[1,1';4',1"]-2'-carboxyterphenylophane

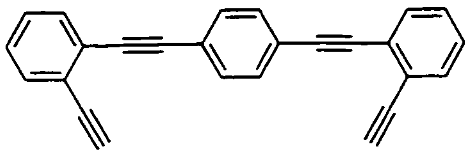


203

Compound **202** (57 mg, 0.17 mmol) was dissolved in THF (2 mL), MeOH (18 mL), H_2O (2 mL) and an excess of LiOH•H_2O was added and stirred for 1 hr. The solution was separated between Et_2O and H_2O and the organic phase washed with 10% HCl and brine, dried and isolated as a yellow solid (15 mg, 27%); m.p. 151-153°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, J = 1.8 Hz, 1H), 7.61 (m, 2H), 7.42 (m, 3H), 7.28 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.5 (s), 150.0 (s), 149.2 (s), 143.1 (s), 142.2 (s), 134.0 (d), 133.0 (d), 132.2 (d), 129.8 (s), 128.8 (d), 128.6 (d), 127.9 (d), 127.3 (d), 127.1 (d), 127.0 (d), 126.9 (d), 126.4 (d), 124.8 (s), 123.1 (s), 98.0 (s), 97.9 (s), 84.8 (s), 83.3 (s); IR ($CHCl_3$) ν = 2999, 2954, 2858, 2169, 1704, 1436, 1213 cm^{-1}; MS (EI) *m/z* 320 (M^+), 274, 248, 222, 162, 83, 70; HRMS calcd for $C_{23}H_{12}O_2$, 320.0837, found 320.0822.

Crystal size 0.20 x 0.20 x 0.20 mm^3, monoclinic, space group $P2_1/c$, scan range $5.3<2\theta<57.28°$, a=12.879(4), b=10.168(3), c=13.466(4) Å, ß=114.545(4)°, V=1604.2(8) Å^3, Z=4, ρ_{calcd}=1.326 g cm-3, u=0.084 mm^{-1}, 3482 unique reflections at -80°C, of which 3482 were taken as observed [$I_o>2.00\sigma(I)$], R=0.0563, R_w=0.0983.(This value is a consequence of the traces of polymer that adhere to the crystal surface.)

1,4-((*o*-Ethynylphenyl)ethynyl)benzene

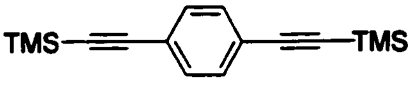


204

From **211**: 1,4-((*o*-Trimethylsilylethynylphenyl)ethynyl)benzene (331 mg, 0.52 mmol) was dissolved in THF (10 mL) and TBAF (1.05 mL, 1.0 M) was added and the resulting solution allowed to stir at RT overnight. When no starting material remained (by TLC) the reaction mixture was separated between ether and water. The ether phase washed with water and brine, dried and evaporated to yield a yellow solid. The solid was further purified by chromatography (Pet Ether) to give the product as a yellow solid (141 mg, 83%)

From **212**: 1,4-((*o*-Trimethylsilylethynylphenyl)ethynyl)benzene (240 mg, 0.51 mmol) was dissolved in a minimum amount of THF (~10 mL) and MeOH (~5 mL) and H_2O (drops) and K_2CO_3 (10-20 equiv.) was added. The solution was stirred vigorously for 1 hour, then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude yellow solid. Purification by chromatography yielded the product as a pale yellow solid (133 mg, 80%); m.p. 105-108°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (s, 4H), 7.52 (dd, J = 7.6, 0.6 Hz, 4H), 7.32 (dt, J = 7.5, 1.6 Hz, 2H), 7.28 (dt, J = 7.5, 1.6 Hz, 2H), 3.37 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.6 (d), 131.8 (d), 131.6 (d), 128.5 (d), 128.1 (d), 126.0 (s), 124.7 (s), 123.2 (s), 93.2 (s), 89.8 (s), 82.1 (s), 81.2 (d); IR ($CHCl_3$): ν = 3303, 3009, 2352, 2333, 2214, 1588, 1513, 1469, 1218 cm^{-1}; MS (EI) *m/z* 326 (M^+), 298, 260, 229, 200, 163, 149, 57, 43; HRMS Calcd for $C_{26}H_{14}$ 326.1096, found 326.1116.

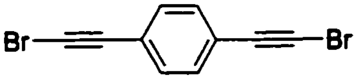
1,4-(Trimethylsilylethynyl)benzene



209

CuI (100 mg, 0.53 mmol), $Pd(PPh_3)_4$ (300 mg, 0.43 mmol) and dibromobenzene (2.00 g, 8.5 mmol) were dissolved in DIPA (30 mL). The resulting dark green solution was degassed for 15 min. by sparging with Ar. Trimethylsilylacetylene (4.6 mL, 32.6 mmol) was added followed by THF (10 mL) to help solubilize the thick mixture. The dark green solution was brought to reflux and heated overnight under Ar. Solvent was then removed under reduced pressure and the resulting black solid was purified by silica flash column chromatography (Pet. Ether) to provide the product as a slightly pale yellow solid (2.20 g, 96%) m.p. 115-117°C; ^{1}H NMR (200 MHz, $CDCl_3$) δ. 7.37 (s, 4H), 0.22 (s, 18H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 131.7 (d), 123,2 (s), 104.0 (s), 95.9 (s), -0.12 (q); IR ($CHCl_3$): ν = 3020, 2955, 2157, 1495, 1251 cm^{-1}; MS (EI) *m/z* 270 (M^+), 255, 225, 183, 143, 120, 73, 44; HRMS Calcd for $C_{16}H_{22}Si_2$ 270.1260, found 270.1260; Anal Calcd for $C_{16}H_{22}Si_2$: C, 71.04; H, 8.20. Found: C, 70.84; H, 8.22.

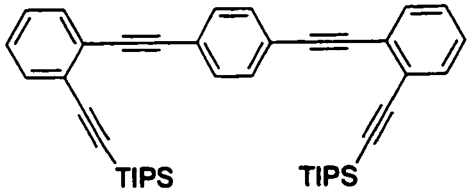
1,4-(Bromoethynyl)benzene



210

1,4-(Trimethylsilylethynyl)benzene (2.01 g, 7.44 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and K_2CO_3 (~30 mg) was added. The solution was stirred vigorously for 1 hour, then separated between pentane and water. The organic phase was washed with water, dried and evaporated on a rotary evaporator until product begun to crystallize. Acetone was added and the solution evaporated until the product begun to crystallize. This procedure was repeated three times in total to remove residual pentane. To a solution of 1,4-ethynylbenzene in acetone was added NBS (3.44 g, 19.4 mmol) and the solution stirred until all NBS had completely dissolved, at which point $AgNO_3$ (~1 crystal) was added and the solution protected from light and stirred overnight (~15 hours). The reaction mixture was then separated between ether and water and the organic phase washed with water and brine, dried and evaporated to yield a crude yellow solid. Purification by chromatography yielded the product as a pale yellow solid (1.91 g, 91%). m.p. >300 °C decomp.; ^1H NMR (200 MHz, $CDCl_3$) δ 7.36 (s, 4H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 131.9 (d), 123.0 (s), 79.5 (s), 52.2 (s); IR ($CHCl_3$): ν = 2195, 1403, 1216, 836 cm^{-1}; MS (EI) *m/z* 282 (M^+), 124, 98, 74, 62; HRMS Calcd for $C_{10}H_4Br_2$ 281.8679, found 281.8696.

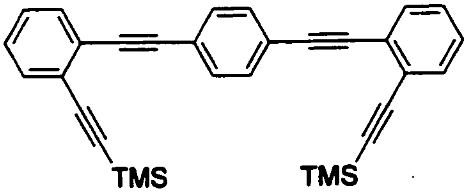
1,4-((*o*-Triisoproplylethynylphenyl)ethynyl)benzene



211

1-Bromo-2-(triispropylsilylethynyl)benzene, **185** (1.04 g, 2.97 mmol) was dissolved in THF (30 mL) and cooled to -78°C and *n*-BuLi (1.3 mL, 2.29 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of $ZnBr_2$ (670 mg, 3.00 mmol) in THF (40 mL) at -78 °C was transferred via canula to the solution of the aryllithium and the solution warmed to 0 °C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (92 mg, 0.08 mmol) and 1,4-(bromoethynyl)benzene (281 mg, 1.00 mmol) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a black semi-solid which was purified by chromatography to give the product as a pale yellow solid (344 mg, 54%). m.p. 97-99°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (m, 4H), 7.48 (s, 4H), 7.27 (m, 4H), 1.11 (s, 42H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.8 (d), 132.0 (d), 131.4 (d), 128.0 (d), 125.9 (s), 125.6 (s), 123.1 (s), 105.3 (s), 95.2 (s), 92.8 (s), 90.0 (s), 18.8 (q), 11.3 (d); IR ($CHCl_3$): ν = 3021, 2959, 2158, 1506, 1469, 1438, 1250 cm^{-1}; MS (FAB) *m/z* 639 (M^++H), 597, 562, 439, 395, 339, 269, 239, 185, 133, 93, 59.

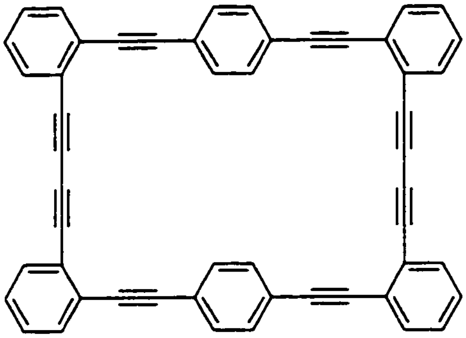
1,4-((*o*-Trimethylsilylethynylphenyl)ethynyl)benzene



212

1-Bromo-2-(trimethylsilylethynyl)benzene, **186** (810.6 mg, 3.18 mmol) in THF (40 mL) was cooled to -78°C and *n*-BuLi (520 μL, 2.04 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of $ZnBr_2$ (720 mg, 3.20 mmol) in THF (10 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (60 mg, 5 mol%) and 1,4-(bromoethynyl)benzene (300mg, 1.06 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid which was purified by chromatography to give the product as a pale yellow solid (245 mg, 49%). m.p. 111-113°C; ^{1}H NMR (500 MHz, CDC13) δ 7.53 (s, 4H), 7.50 (dd, J = 6.3, 4.5 Hz, 4H), 7.27 (m, 4H), 0.26 (s, 18H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.4 (d), 131.7 (d), 131.6 (d), 128.2 (d), 128.1 (d), 125.8 (s), 125.8 (s), 123.3 (s), 103.4 (s), 98.8 (s), 93.1 (s), 92.8 (s), 90.2 (s), 0.005 (q); IR ($CHCl_3$): ν = 3021, 2959, 2158, 1506, 1469, 1438, 1250 cm^{-1}; MS (FAB) *m/z* 471 (M^++H), 391, 246, 185, 132, 73.

3,9-Di-*o*-phenyl-1,5,7,11-tetrayne-[12.12]paracyclophane

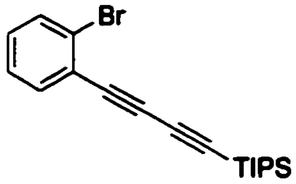


213

$Cu(OAc)_2$ (260 mg, 2.0 mmol) was dissolved in pyridine: ether (3:1) (145 mL) at RT and 1,4-((*o*-ethynylphenyl)ethynyl)benzene (112 mg, 0.34 mmol) in 20 mL of pyridine:ether (3:1) was added over 3 hr *via* syringe pump. The solution was protected from light and left to stir at RT for 3 days. The solution was then poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl (until all pyridine was removed), saturated $NaHCO_3$ (aq) and brine, dried and evaporated to yield a yellow solid. The solid was further purified by chromatography (6:1 Hexanes: CH_2Cl_2) to yield the product as a yellow solid (46.5 mg, 21%). m.p. >300 °C decomp.; ^1H NMR (500 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 7.5, 0.8 Hz, 4H), 7.50 (dd, *J* = 7.5, 0.8 Hz, 4H), 7.32 (m, 8H), 7.29 (s, 8H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.3 (d), 131.6 (d), 131.4 (d), 129.0 (d), 128.1 (d), 127.5 (s), 124.8 (s), 122.7 (s), 94.5 (s), 89.2 (s), 81.2 (s), 78.1 (s); IR ($CHCl_3$): ν = 2214, 2156, 1719, 1506, 1467, 1442 cm^{-1}; MS (FAB) *m/z* 672, 550, 454, 391, 279, 168, 93, 55.

Crystal size 0.10 x 0.30 x 0.40 mm^3, orthorhombic, space group *Pbcn*, scan range $1.33<\theta<28.74°$, a=30.657(3), b=8.5590(7), c=13.7917(12) Å, ß=90°, *V*=3618.9 $Å^3$, Z=4, ρ_{calcd}=1.191 g cm-3, u=0.068 mm^{-1}, 4437 unique reflections at -80°C, of which 28287 were taken as observed $[I_O>2.00\sigma(I)]$, R=0.0960, R_W=1203.

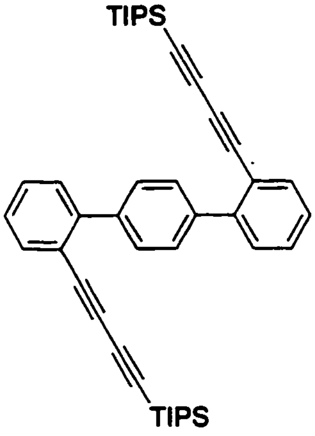
1-Bromo-2-(1-triispropylsilyl-1,3-butadiynyl)benzene



215

CuI (141 mg, 0.74 mmol), $Pd(PPh_3)_2Cl_2$ (142 mg, 0.20 mmol) and bromoiodobenzene (3.43 g, 12.1 mmol) were dissolved in DEA (50 mL). The resulting dark green solution was degassed for 15 min. by sparging with Ar. 1-Triisopropylsilyl-1,3-butadiyne (3.7 g, 17.9 mmol) was added followed by THF (50 mL) to help dissolve the thick mixture. The dark green solution was brought to reflux and heated overnight under Ar. Solvent was then removed under reduced pressure and the resulting black solid was purified by chromatography (Pet. Ether) to provide the product as a slightly pale yellow liquid (1.52 g, 35%); ^{1}H NMR (200 MHz, $CDCl_3$) 7.54 (m, 2H), 7.22 (m, 2H), 1.11 (s, 21H); ^{13}C NMR (50 MHz, $CDCl_3$) 134.7 (d), 132.5 (d), 130.1 (d), 126.9 (s), 126.2 (s), 124.1 (s), 89.8 (d), 89.2 (d), 79.5 (s), 73.5 (s), 18.5 (q), 11.3 (d); IR ($CHCl_3$): ν = 2946, 2867, 2205, 2101, 1465, 1434, 1385, 1296, 1064, 1009.7, 910, 883 cm^{-1}; MS (EI) *m/z* 362 (M^+), 360, 320, 319, 317, 291, 289, 277, 275, 263, 261, 249, 247, 233, 209, 181, 167, 153, 131, 109, 91, 77, 53, 43; HRMS Calcd for $C_{19}H_{25}BrSi$ 360.0910, found 360.0922; Anal Calcd for $C_{19}H_{25}BrSi$: C, 63.15; H, 6.98. Found: C, 62.95; H, 6.91.

2,2''-Bis-(4-triisopropylsilanyl-buta-1,3-diynyl)-[1,1';4',1'']terphenyl



216

Compound **215** (740 mg, 2.04 mmol) in THF (30 mL) was cooled to -78°C and *n*-BuLi (890 μL, 2.04 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of ZnBr$_2$ (455 mg, 2.04 mmol) in THF (40mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of Pd(PPh$_3$)$_4$ (70 mg, 0.060 mmol) and 1,4-dibromobenzene (152 mg, 0.64 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a pale yellow solid (92 mg, 23%). m.p. 126-27°C; ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.69 (s, 4H), 7.65 (dd, *J* = 7.7, 0.9 Hz, 2H), 7.44 (dt, *J* = 6.4, 1.2 Hz, 2H), 7.41 (dt, *J* = 7.0, 1.3 Hz, 2H), 7.29 (dt, *J* = 7.0, 1.0 Hz, 2H), 1.06 (s, 42H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 144.7 (s), 139.2 (s), 134.7 (d), 129.6 (d), 129.3 (d), 128.8 (d), 127.0 (d), 119.8 (s), 89.7 (s), 88.3 (s), 77.6 (s), 75.3 (s), 18.5 (q), 11.2 (d); IR (CHCl$_3$): ν = 2956, 2096, 1460, 1068, 658 cm^{-1}; MS (EI) *m/z* 638 (M$^+$), 595, 553, 511, 469, 395, 367, 276, 248, 206, 157, 115, 87, 73, 59, 43; HRMS Calcd for C$_{44}$H$_{54}$Si$_2$ 638.3766, found 638.3772.

Synthesis of Diynes and Tetraynes by an *in-situ* Desilylation/Dimerization Strategy.

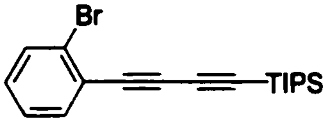
Standard Desilylation/Oxidative Dimerization Method Using Cu(OAc)$_2$

A solution of tetra-*n*-butylammonium fluoride (1.0 M in THF, 1 eq) in THF (5 mL) was added over 2 h *via* syringe pump to a stirred solution of Cu(OAc)$_2$ (3 eq) and corresponding TIPS-acetylene (1 eq) in pyridine/ether (3:1) (substrate concentration was 3.3 mM). The blue solution became emerald green once addition began. Once addition was complete, the solution was poured into ether and HCl (1 M). The organic phase was washed excessively with HCl (1 M) until all pyridine was removed and the organic phase was dried and concentrated to yield a crude solid. The solid was further purified by chromatography (petroleum ether unless otherwise specified) to yield the dimer product.

Standard Desilylation/Oxidative Dimerization Method Using CuCl/TMEDA

A solution of tetra-*n*-butylammonium fluoride (1.0 M in THF, 1 eq) in THF (5 mL) was added over 2 h *via* syringe pump to a solution of CuCl (3 eq), TMEDA (100 eq) and corresponding TIPS-acetylene (1 eq) in benzene (substrate concentration was 3.3 mM). The lime-green solution became emerald green after addition began. Once addition was complete, the solution was poured into ether and HCl (1 M). The organic phase was washed to remove TMEDA and the organic phase was dried and concentrated to yield a crude solid. The solid was further purified by chromatography (petroleum ether unless otherwise specified) to yield the dimer product.

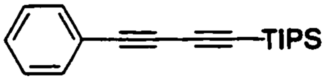
[4-(2-Bromophenyl)-buta-1,3-diynyl]-triisopropyl-silane



215

*n*BuLi (36.4 mL, 82.6 mmol) was added to a solution of **160**[159] (10 g, 41.3 mmol) in THF (60 mL) at 0 °C and the resulting deep yellow solution was stirred for 5 min. A solution of $ZnBr_2$ (9.3 mg, 41.3 mmol) in THF (20 mL) at 22 °C was transferred by canula to the solution and warmed to 22 °C and stirred for 15 min. A solution of $Pd(PPh_3)_4$ (600 mg, 0.52 mmol) and 1-bromo-2-iodobenzene (7.8 g, 27.5 mmol) was then transferred by canula to the reaction mixture and heated to reflux overnight (~15 h). The reaction was then quenched by the addition of silica gel. Concentration and dry packed chromatography give the product as a clear liquid (3.05 g, 35%); ^1H NMR (200 MHz, $CDCl_3$) δ 7.54 (m, 2H), 7.22 (m, 2H), 1.11 (s, 21H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 134.7 (d), 132.5 (d), 130.1 (d), 126.9 (s), 126.2 (s), 124.1 (s), 89.8 (d), 89.2 (d), 79.5 (s), 73.5 (s), 18.5 (q), 11.3 (d); MS (EI) *m/z* 360 (M^+), 320, 319, 317, 291, 289, 277, 275, 263, 261, 209, 153; HRMS calcd for $C_{19}H_{25}BrSi$ 360.0910 (M^+), found 360.0922; Anal calcd for $C_{19}H_{25}BrSi$: C 63.15, H 6.98, found C 62.95, H 6.91.

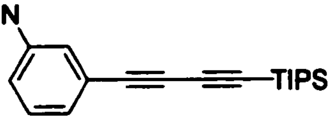
Triisopropyl-(4-phenyl-buta-1,3-diynyl)-silane



218

n-BuLi (2.27 M, 15.8 mL, 35.8 mmol) was added to a solution of **160**[159] (4.34 g, 17.9 mmol) in THF (40 mL) at –78 °C and the resulting deep yellow solution was stirred for 5 min. A solution of $ZnBr_2$ (4.12 g, 18.3 mmol) in THF (40 mL) at 22 °C was transferred by canula to the solution and warmed to 22 °C and stirred for 15 min. A solution of $Pd(PPh_3)_4$ (500 mg, 5 mol%) and iodobenzene (1.0 mL, 8.94 mmol) was transferred by canula to the solution which was then heated to reflux overnight (~18 h). The reaction mixture was then quenched by the addition of silica gel. Concentration and dry packed chromatography give the product as a yellow oil (1.61 g, 64%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 8.2, 1.3 Hz, 2H), 7.37 – 7.29 (m, 3H), 1.13 (s, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.6 (d), 129.2 (d), 128.3 (d), 121.5 (s), 89.6 (s), 87.8 (s), 75.5 (s), 74.7 (s), 18.5 (q), 11.3 (d); MS (EI) *m/z* 282 (M^+), 239, 211, 183, 169; HRMS calc'd for $C_{19}H_{26}Si$ 282.1804 (M^+), found 282.1787.

[4-(3-Dibutylamino-phenyl)-buta-1,3-diynyl]-triisopropyl-silane



223

A solution of *n*BuLi (2.04 M, 22.4 mL, 45.6 mmol) was added to a –78 °C solution of **160**[159] (5.50 g, 22.7 mmol) in THF (150 mL). The resulting pale yellow colored solution was stirred for 2 min followed by the addition of a solution of $ZnBr_2$ (5.27 g, 23.4 mmol, 1.55 eq) in THF (100 mL). The colourless solution was stirred at –78 °C for 5 min then warmed to 0 °C for 15 min. A mixture of *N,N*-dibutyl-3-iodoaniline (5.00 g, 15.1 mmol, 1 eq), $Pd(PPh_3)_4$ (1.75 g, 10 mol%) in THF (150 mL) was added by canula and the reaction was heated to reflux for 18 h. Once cooled to 22 °C, silica gel was added to the reaction flask and its contents were concentrated to dryness. After chromatography (petroleum ether / CH_2Cl_2, 20:1), the product was isolated as a yellow oil (4.70 g, 76%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (t, *J* = 7.9 Hz, 1H), 6.79 – 6.76 (m, 3H), 3.24 (t, *J* = 7.7 Hz, 4H), 1.59 – 1.52 (m, 4H), 1.40 – 1.30 (m, 4H), 1.14 (s, 21H), 0.97 (t, *J* = 7.3 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.9 (s), 129.1 (d), 121.9 (s), 119.7 (d), 115.3 (d), 113.0 (d), 90.0 (s), 86.7 (s), 73.3 (s), 65.8 (s), 50.7 (t), 29.0 (t), 20.4 (t), 18.4 (q), 13.9 (q),

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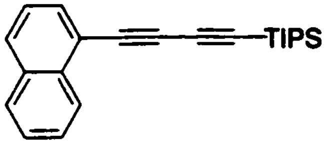
[159] Lu, Y.F.; Harwig, C.W.; Fallis, A.G. *J. Org. Chem.* **1993**, *58*, 4202.

11.3 (d); MS (EI) *m/z* 409 (M^+), 319, 221, 163, 135; HRMS calc'd for $C_{27}H_{43}NSi$ 409.3165 (M^+), found 409.3157; Anal. calc'd for $C_{27}H_{43}NSi$: C 79.15, H 10.58, found C 78.95, H 10.68.

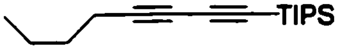
Triisopropyl-(4-naphthalen-1-yl-buta-1,3-diynyl)-silane



225

*n*BuLi (1.45 µL, 2.28 M, 3.30 mmol) was added to a solution of **160**[2] (400 mg, 1.65 mmol) in THF (15 mL) at 0 °C and the resulting deep yellow solution was stirred for 5 min. A solution of $ZnBr_2$ (340 mg, 1.65 mmol) in THF (8 mL) at 22 °C was transferred by canula to the solution and warmed to 22 °C and allowed to stir for 15 min. A solution of $Pd(PPh_3)_4$ (50 mg, 0.04 mmol) and 1-bromonaphthalene (310 mg, 1.50 mmol) was then transferred by canula to the solution and heated to reflux overnight (~15 h). The reaction mixture was then quenched by the addition of silica gel. Concentration and dry packed chromatography gave the product as a pale yellow solid (350 mg, 64%); ^1H NMR (200 MHz, $CDCl_3$) δ 8.38 (d, *J* = 7.9, 1H), 7.80 (m, 2H), 7.37-7.63 (m, 4H), 1.20 (s, 21H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 134.0 (s) 133.0 (s), 132.3 (d), 129.7 (d), 128.4 (d), 127.1 (d), 126.6 (d), 126.0 (d), 125.1 (d), 119.1 (s), 89.7 (s), 88.9 (s), 79.3 (s), 73.9 (s), 18.6 (q), 11.3 (d); MS (EI) *m/z* 332 (M^+), 289, 247, 219, 203, 184, 141, 115, 44; HRMS calc'd for $C_{23}H_{28}Si$ 332.1961 (M^+), found 332.1955.

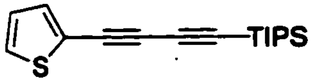
Triisopropyl-octa-1,3-diynyl-silane



227

1-Hexyne (500 mg, 6.09 mmol), $Pd_2(dba)_3$ (70 mg, 0.76 mmol), CuI (20 mg, 0.11 mmol), 1,2,2,6,6-pentamethylpiperidine (1.37 mL, 7.6 mmol) and 1-bromo-2-(triisopropylsilyl)acetylene (1.0 g, 0.38 mmol) were combined in benzene (10 mL) at 22 °C. The solution was stirred for 19 h. The reaction mixture was quenched by the addition of silica gel. Concentration and dry packed chromatography gave the product as a pale yellow liquid (433 mg, 43%); ^1H NMR (200 MHz, $CDCl_3$) δ 2.25 (t, *J* = 6.7, 2H) 1.48 (m, 2H), 1.05 (s, 21H), 1.05 (m, 2H), 0.88 (t, *J* = 6.8, 3H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 90.1 (s), 79.7 (s), 78.7 (s), 65.7 (s), 30.1 (t), 21.9 (t), 18.9 (q), 18.4 (q), 13.5 (t), 11.2 (d); MS (EI) *m/z* 262 (M^+), 219, 191, 177, 163, 149, 111, 97, 83, 69, 44; HRMS calc'd for $C_{17}H_{30}Si$ 262.2118 (M^+), found 262.2126.

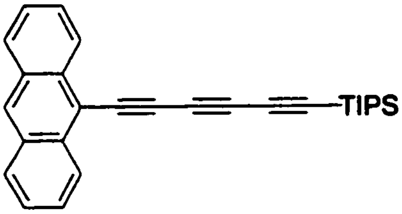
Triisopropyl-(4-thiophen-2-yl-buta-1,3-diynyl)-silane



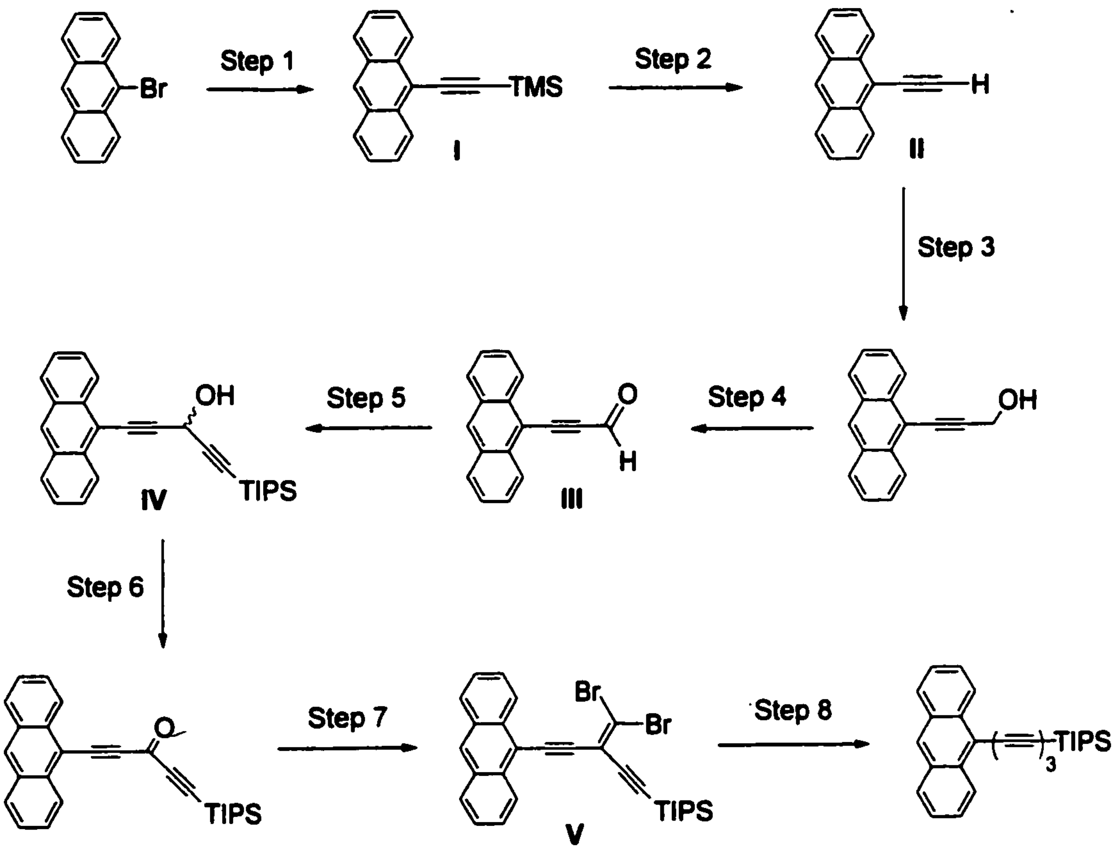
229

n-BuLi (2.06 mL, 3.3 mmol) was added to a solution of **160**[2] (400 mg, 1.65 mmol) in THF (15 mL) at 0 °C and the resulting deep yellow solution was stirred for 5 min. A solution of $ZnBr_2$ (375 mg, 1.65 mmol) in THF (10 mL) at 22 °C was transferred by canula to the solution and warmed to 22 °C and allowed to stir for 15 min. A solution of $Pd(PPh_3)_4$ (50 mg, 0.04 mmol) and 2-bromothiophene (110 μL, 1.14 mmol) was then transferred by canula to the solution and heated to reflux overnight (~15 h). The reaction mixture was then quenched by the addition of silica gel. Concentration and dry packed chromatography gave the product as a yellow oil (284 mg, 86%); ^1H NMR (200 MHz, $CDCl_3$) δ 7.27 (m, 2H), 6.93 (m, 1H), 1.10 (s, 21H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 134.5 (d), 128.6 (d), 127.0 (d), 121.7 (s), 90.2 (s), 89.2 (s), 78.7 (s), 68.6 (s), 18.5 (q), 11.2 (d); MS (EI) *m/z* 288 (M^+), 245, 217, 189, 165, 135, 94, 59; HRMS calc'd for $C_{17}H_{24}SSi$ 288.1361 (M^+), found 288.1368.

(6-Anthracen-9-yl-hexa-1,3,5-triynyl)-triisopropyl-silane



231



Step 1: 4-Bromoanthracene (1.5 g, 5.83 mmol), $Pd(PPh_3)_2Cl_2$ (100 mg, 0.14 mmol), Et_3N (30 mL), and CuI (50, mg, 0.26 mmol) were combined in THF (30 mL). The solution was degassed by passing a stream of argon gas through the solution for 10 min. TMS-acetylene was then added (905 uL, 6.42 mmol) and the mixture heated at reflux for 15 h. The reaction mixture was cooled to 22 °C and quenched by the addition of silica gel. Concentration and dry packed chromatography (petroleum ether) give anthracen-9-ylethynyl-trimethylsilane (**I**) as a yellow solid (1.58 g, 100%). The silylacetylene was carried on directly to the next step.

Step 2: Compound **I** (890 mg, 3.24 mmol) and K_2CO_3 (excess) were combined in THF (15 mL) and MeOH (15 mL). The mixture was vigorously stirred for 8 h and poured into a mixture of ether and water. The organic phase was dried and concentrated and the crude solid was passed through a silica gel plug (petroleum ether) to yield 9-ethynyl-anthracene (**II**) as a yellow solid (650 mg, 99%). The acetylene was carried on directly to the next step.

Step 3: *n*-BuLi (1.35 mL, 2.27 M, 3.04 mmol) was added to a solution of 9-ethynylanthracene (**II**) (590 mg, 2.92 mmol) in THF (20 mL) and was cooled to –78 °C and stirred for 15 min. A bright yellow solution resulted. The reaction was quenched by the addition of *p*-formaldehyde (262 mg, 2.91 mmol) in THF (20 mL) and stirred at 22 °C for 15 h. The mixture was partitioned between ether and water. The organic phase was dried, concentrated, and chromatographed (petroleum ether/ether, 5:1) to yield 3-anthracen-9-yl-prop-2-yn-1-ol, **233**, as a yellow-brown solid (600 mg, 89%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.50 (d, *J* = 8.9, 2H), 8.40 (s, *J* = 7.8 Hz, 1H), 7.97 (d, *J* = 7.8 Hz, 2H), 7.54 (m, 4H), 4.81 (s, 2H), 2.03 (br s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 132.7 (s), 130.9 (s), 128.6 (d), 127.9 (d), 126.6 (d), 126.5 (d), 125.6 (d), 108.6 (s), 98.3 (s), 82.3 (s), 52.1 (t); MS (EI) *m/z* 232 (M^+), 202, 178, 143, 101, 44, 29; HRMS calc'd for $C_{17}H_{12}O$ 232.0887 (M^+), found 232.0896.

Step 4: The alcohol was then oxidized in the following manner. $BaMnO_4$ (2.8 g, 10.9 mmol) was added to a solution of 3-anthracen-9-yl-prop-2-yn-1-ol, **233**, (490 mg, 2.11 mmol) in methylene chloride (15 mL) and the mixture was stirred for 4 h. The mixture was then filtered through a pad of Celite™ and the filtrate was concentrated to yield anthracen-9-yl-propynal (**III**) as a yellow solid (482 mg, 99%), which was carried on directly to the next step.

Step 5: Alkynylation was achieved as follows. *n*-BuLi (1.7 mL, 2.27 M, 3.86 mmol) was added to a 0 °C solution of TIPS-acetylene (875 μL, 3.91 mmol) in THF (10 mL) and was stirred at 0 °C for 1 h. Anthracen-9-yl-propynal (**III**) (600 mg, 2.61 mmol) was added and the reaction mixture was to warmed to 22 °C and stirred for 15 h. The mixture was partitioned between ether and water. The organic phase was dried, concentrated, chromatographed (petroleum ether/ ether, 5:1) to yield 1-anthracen-9-yl-5-triisopropylsilanyl-penta-1,4-diyn-3-ol (**IV**) as an orange oil (415 mg, 39%). Alcohol **IV** was carried on directly to the next step.

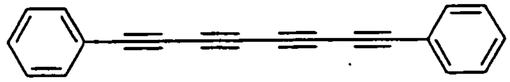
Step 6: Alcohol **IV** (415 mg, 1.01 mmol) was immediately added to a mixture of PCC (260 mg, 1.20 mmol), Celite™ (400 mg) in methylene chloride (15 mL) and stirred at 22 °C for 5 h. The mixture was then filtered through a pad of Celite™ and the filtrate evaporated to yield 1-anthracen-9-yl-5-triisopropylsilanyl-penta-1,4-diyn-3-one, **234**, as a yellow oil (200 mg, 48%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.2 Hz, 2H), 8.38 (s, 1H), 7.89 (d, *J* =8.4 Hz, 2H), 7.47 (m, 4H), 1.20 (s, 21H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 159.9 (s), 134.5 (s), 132.0 (d), 130.6 (s), 128.9 (d), 128.0 (d), 126.0 (d), 125.8 (d), 112.1

(s). 105.5 (s), 100.9 (s), 97.1 (s), 89.7 (s); MS (EI) *m/z* 410 (M^+), 382, 339, 297, 269, 229, 208, 180, 143, 79, 61, 44; HRMS calc'd for $C_{28}H_{30}OSi$ 410.2067 (M^+), found 410.2052.

Step 7: Ketone, **234** (170 mg, 0.41 mmol), CBr_4 (194 mg, 0.59 mmol), and PPh_3 (333 mg, 1.26 mmol) were combined in methylene chloride (40 mL) and stirred for 15 h. Pentane was then added to the mixture and a white precipitate formed ($Ph_3P{=}O$). The mixture was filtered and the filtrate evaporated to yield a yellow oil. This oil was dissolved in a minimum amount of methylene chloride and diluted with pentane to precipitate any remaining triphenylphosphine oxide. This sequence was repeated until the addition of pentane produced no visible precipitation. The dibromide, (5-Anthracen-9-yl-3-dibromomethylene-penta-1,4-diynyl)-triisopropyl-silane (V), was isolated as a pale yellow oil (150 mg, 64%). The dibromide was carried on directly to the next step.

Step 8: *n*BuLi (235 uL, 2.27 M, 0.53 mmol) was added to a –78 °C solution of dibromide V (150 mg, 0.26 mmol) in hexanes (10 mL) and the reaction mixture was slowly warmed to -40 °C. The reaction was quenched by adding $NH_4Cl_{(aq)}$ and partitioned between ether and water. The organic phase was dried and concentrated to yield **231** as a bright yellow solid (42 mg, 41%); Mp: 124 – 126 °C; ^1H NMR (200 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.42 (d, J = 3.8Hz, 2H), 7.97 (d, J =8.0 Hz, 2H), 7.54 (m, 4H), 1.12 (s, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.7 (s), 130.9 (s), 129.4, (d), 128.9 (d), 127.5 (d), 126.3 (d), 125.9 (d), 114.4 (s), 89.8 (s), 88.5 (s), 85.1 (s), 74.0 (s), 70.4 (s), 60.8 (s), 18.5 (q), 11.3 (d); MS (EI) *m/z* 406 (M^+), 363, 321, 293, 253, 153, 112, 70; HRMS calc'd for $C_{29}H_{30}Si$ 406.2118 (M^+), found 406.2100.

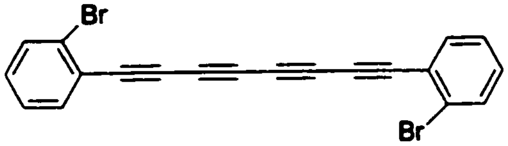
1,8-Diphenyl-octa-1,3,5,7-tetrayne



220

Compound **218** (100 mg, 0.35 mmol), $Cu(OAc)_2$ (192 mg, 1.05 mmol) and TBAF (1.0 M in THF, 355 µL) were combined in a mixture of pyridine/diethyl ether (3:1, 100 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as a yellow solid (40 mg, 91%); ^1H NMR (500 MHz, $CDCl_3$) δ 7.54 – 7.51 (m, 2H), 7.42 – 7.37 (m, 1H), 7.36 – 7.32 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.2 (d), 130.0 (d), 128.5 (d), 120.5 (s), 77.7 (s), 74.4 (s), 67.1 (s), 63.6 (s); MS (EI) *m/z* 250 (M^+), 226, 162, 125; HRMS calc'd for $C_{20}H_{10}$ 250.0783 (M^+), found 250.0784.

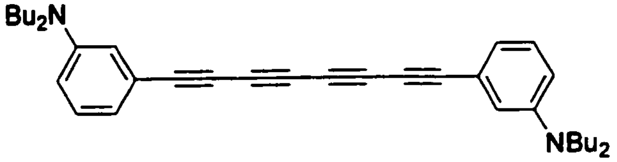
1,8-Di-(*o*-bromophenyl)-octa-1,3,5,7-tetrayne



222

Compound **215** (500 mg, 1.58 mmol), $Cu(OAc)_2$ (856 mg, 4.73 mmol) and TBAF (1.0 M in THF, 1.58 mL) were combined in pyridine/diethyl ether (3:1, 500 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as a pale yellow solid (232 mg, 92%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, J= 0.8 Hz, 1H), 7.54 (m, 1H), 7.28-7.19 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.1 (d), 132.7 (d), 130.9 (d), 127.2 (d), 126.8 (s), 123.1 (s), 78.3 (s), 76.0 (s), 68.5 (s), 64.0 (s); MS (EI) *m/z* 407 (M^+), 383, 359, 303, 267, 248, 224, 198, 112, 75, 39; HRMS calc'd for $C_{20}H_8Br_2$ 405.8992 (M^+), found 405.9010. X-ray structural analyses for compound **222**: Crystal size 0.22 x 0.18 x 0.07 mm^3, triclinic, space group *P*-1, scan range $2.92 < 2\theta < 28.69°$, a = 3.9243(6), b = 7.1868(12), c = 14.354(2) Å, ß = 103.008(3)°, V = 392.01(11) Å^3, Z = a, ρcalcd = 1.729 g cm^{-3}, u = 5.162 mm^{-1}, 1771 unique reflections at –80 °C, of which 3059 were taken as observed [$I_o > 2.00\sigma(I)$], R = 0.0530, R_W = 0.1137. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-163717 (**2d**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

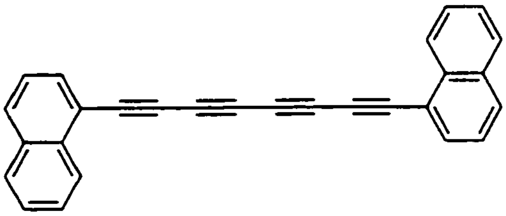
1,8-Di-(*m*-dibutylaminophenyl)-octa-1,3,5,7-tetrayne



224

Compound **223** (100 mg, 0.24 mmol), $Cu(OAc)_2$ (133 mg, 0.73 mmol) and TBAF (1.0 M in THF, 100 µL) were combined in pyridine/diethyl ether (3:1, 100 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as a yellow oil (50 mg, 82%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.12 (t, *J* = 8.0 Hz, 2H), 6.81 – 6.67 (m, 6H), 3.22 (t, *J* = 7.5 Hz, 8H), 1.63 – 1.42 (m, 8H), 1.42 – 1.22 (m, 8H), 0.97 (t, *J* = 7.1 Hz, 12H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 147.8 (s), 129.3 (d), 120.8 (s), 120.0 (d), 115.4 (d), 113.6 (d), 79.0 (s), 73.1 (s), 66.8 (s), 63.7 (s), 50.6 (t), 29.2 (t), 20.3 (t), 14.0 (q); MS (EI) *m/z* 506 (M^+), 461, 419, 229, 186; HRMS calc'd for $C_{36}H_{44}N_2$ 506.3504 (M^+), found 506.3586.

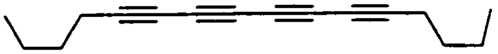
1,8-Di-(1-naphthyl)-octa-1,3,5,7-tetrayne[102]



226

Compound **225** (150 mg, 0.45 mmol), $Cu(OAc)_2$ (245 mg, 1.36 mmol) and TBAF (1.0 M in THF, 451 µL) were combined in pyridine/diethyl ether (3:1, 136 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as a bright yellow solid (76 mg, 96%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.9, 2H), 7.86 (m, 4H), 7.37-7.68 (m, 8H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 134.2 (s) 133.3 (d), 133.0 (s), 130.6 (d), 128.6 (d), 127.4 (d), 126.8 (d), 125.9 (d), 125.1 (d), 118.1 (s), 79.03 (s), 79.0 (s), 77.7 (s), 76.5 (s); MS (EI) *m/z* 350 (M^+), 326, 302, 279, 149, 85, 71, 29; HRMS calc'd for $C_{28}H_{14}$ 350.1096 (M^+), found 350.1095.

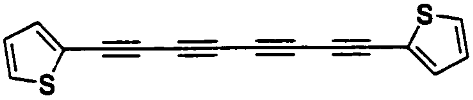
Hexadeca-5,7,9,11-tetrayne



228

Compound **227** (120 mg, 0.46 mmol), $Cu(OAc)_2$ (250 mg, 1.37 mmol) and TBAF (1.0 M in THF, 460 µL) were combined in pyridine/diethyl ether (3:1, 140 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as a clear liquid (48 mg, 98%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.25 (t, *J* = 6.7, 2H), 1.48 (m, 2H), 1.05 (s, 21H), 1.05 (m, 2H), 0.88 (t, $J = 6.8$, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 80.3 (s), 65.6 (s), 61.3 (s), 60.5 (s), 29.9 (t), 21.8 (t), 19.1 (t), 13.4 (t); MS (EI) *m/z* 210 (M^+), 165, 152, 80, 29; HRMS calc'd for $C_{16}H_{18}$ 210.1409 (M^+), found 210.1429.

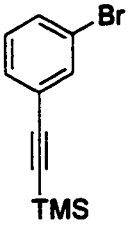
1,8-Di-(2-thiophenyl)-octa-1,3,5,7-tetrayne[103]



230

Compound **229** (90 mg, 0.31 mmol), $Cu(OAc)_2$ (375 mg, 0.93 mmol) and TBAF (1.0 M in THF, 700 μL) were combined in pyridine/diethyl ether (3:1, 200 mL) at 22 °C as per the standard method. Chromatography yielded the title compound as bright yellow crystals (33 mg, 82%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.39 (dd, J= 5.7, 4.5 Hz, 1H), 7.32 (t, J= 4.5 Hz, 1H), 6.97 (t, J= 4.5 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 136.2 (d), 130.1 (d), 127.3 (d), 120.9 (d), 78.8 (s), 71.3 (s), 64.6 (s), 61.6 (s); MS (EI) *m/z* 238 (M^+), 214, 169, 150, 122, 69, 29; HRMS calc'd for $C_{14}H_6S_2$ 237.9911(M^+ - C_2), found 237.9897.

(3-Bromo-phenylethynyl)-trimethyl-silane[31,32,33]

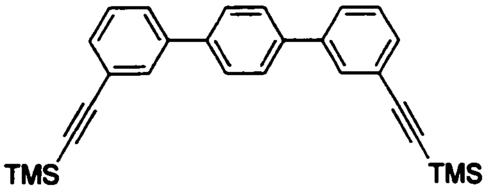


236

CuI (43 mg, 0.22 mmol), $Pd(PPh_3)_2Cl_2$ (52 mg, 0.07 mmol) and **235** (1.00 g, 3.53 mmol) were added to a flame dried round bottom flask and dissolved in DEA (15 mL) and THF (10 mL). The resulting dark green solution was degassed for 15 minutes by spargingwith Ar or N_2. Trimethylsilylacetylene (0.54 mL, 3.82 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide a slightly pale yellow liquid (850 mg, 95%); the product as a slightly pale yellow liquid (5.94 g, 91%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (t, J = 1.5 Hz, 1H), 7.41 (m, 2H), 7.13 (dt, J = 7.8, 1.5 Hz, 1H), 0.24 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.7 (d), 131.6 (d), 130.4 (d), 129.6 (d), 125.1 (s), 122.0 (s), 103.3 (s), 95.8 (s), -0.15 (q); IR (neat): ν = 2960, 2163, 1655, 1559, 1403, 1250, 872 cm^{-1}; MS (EI) *m/z*

254, 252 (M^+), 239, 222, 208, 179, 158, 143, 115, 69, 32; HRMS calcd for $C_{11}H_{13}BrSi$ 251.9970, found 251.9894; Anal Calcd for $C_{11}H_{13}SiBr$: C, 52.18; H, 5.18. Found: C, 51.95; H, 4.90.

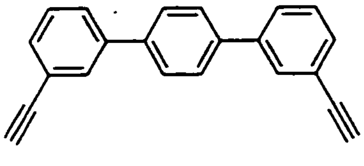
1,4-(*m*-Trimethylsilylethynylphenyl)benzene



237

1-Bromo-3-(trimethylsilylethynyl)benzene, **236** (400 mg, 1.57 mmol) in THF (10 mL) was cooled to -78°C and *n*-BuLi (773 μL, 1.57 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (353 mg, 1.57 mmol) in THF (5 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (100 mg, 0.086 mmol) and 1,4-dibromobenzene (125 mg, 0.53 mmol) in THF (5 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a colourless oil (62 mg, 83%).; ^1H NMR (500 MHz, CDCl3) δ 7.74 (t, *J* = 1.5 Hz, 2H), 7.64 (s, 4H), 7.56 (dt, *J* = 7.7, 1.7 Hz, 2H), 7.45 (dt, *J* = 7.7, 1.3 Hz, 2H), 7.37 (t, J = 7.7 Hz, 2H), 0.26 (s, 18H); ^{13}C NMR (125 MHz, CDCl3) δ 140.6 (s), 139.5 (s), 130.8 (d), 130.6 (d), 128.7 (d), 127.5 (d), 127.2 (s), 123.7 (s), 105.0 (s), 94.4 (s), -0.021 (q); IR (CHCl3): ν = 2965, 2156, 1635, 1560, 1242, 841 cm^{-1}; MS (EI) *m/z* 422 (M^+), 366, 331, 277, 245, 218, 173, 149, 115, 73; HRMS Calcd for $C_{28}H_{30}Si_2$ 422.1887, found 422.1892.

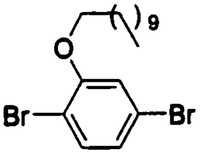
1,4-(*m*-Ethynylphenyl)benzene



238

Compound **237** (180 mg, 0.42 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a white solid (117 mg, 99%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (t, *J* = 1.6 Hz, 2H), 7.64 (s, 4H), 7.60 (dt, *J* = 7.8, 1.6 Hz, 2H), 7.47 (dt, *J* = 7.7, 1.4 Hz, 2H), 7.40 (t, J = 7.7 Hz, 2H), 3.10 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.8 (s), 139.5 (s), 131.0 (d), 130.7 (d), 128.9 (d), 127.5 (d), 127.5 (s), 122.7 (s), 83.56 (s), 77.4 (d); IR ($CHCl_3$): *v* = 3290, 2929, 2156, 1652, 1635, 1390, 1255, 792 cm^{-1}; MS (EI) *m/z* 278 (M^+), 231, 202, 162, 149, 112, 57; HRMS Calcd for $C_{22}H_{14}$ 278.1096, found 278.1089.

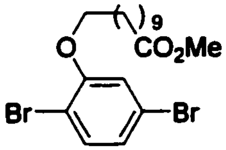
1,4-Dibromo-2-undecyloxy-benzene



258

Compound **257** (2.40 g, 9.52 mmol) and 1-bromoundecane (2.34 mL, 9.97 mmol) were dissolved in refluxing acetone (60 mL) and an excess of K_2CO_3 was added and stirred overnight (~15 hr). Silica gel was added to the solution and the solvent evaporated under reduced pressure. The crude solid was then purified by silica gel chromatography (Pet. Ether) to yield the product as a colourless oil (3.8 g, 99%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.8 Hz, 1H), 6.96 (dd, *J* = 8.8, 2.1 Hz, 1H), 6.90 (d, *J* = 2.1 Hz, 1H), 3.97 (t, *J* = 6.4 Hz, 2H), 1.84 (m, 2H), 1.19 – 1.52 (m, 16H), 0.87 (t, *J* = 6.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 156.2 (s), 134.0 (d), 124.5 (d), 121.4 (s), 116.6 (d), 111.1 (s), 69.5 (t), 32.9 (t), 31.9 (t), 29.6 (t), 29.4 (t), 29.3 (t), 28.8 (t), 28.2 (t), 25.9 (t), 22.7 (t), 14.1 (q); IR (neat): ν = 2924, 2854, 1559, 1457, 1252, 1035 cm^{-1}; MS (EI) 324 (M$^+$-Br), 280, 234, 219, 169, 135, 97, 85, 69, 43.

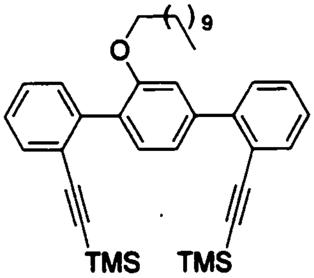
11-(2,5-Dibromo-phenoxy)-undecanoic acid methyl ester



259

Compound **257** (1.10 g, 4.36 mmol) and methyl 11-bromoundecanoate (1.34 g, 4.80 mmol) were dissolved in refluxing acetone (30 mL) and an excess of K_2CO_3 was added and stirred overnight (~15 hr). Silica gel was added to the solution and the solvent evaporated under reduced pressure. The crude solid was then purified by silica gel chromatography (Pet. Ether) to yield the product as a fluffy white solid (1.81 g, 92%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.35 (d, J = 8.8 Hz, 1H), 6.96 (dd, J = 8.8, 2.1 Hz, 1H), 6.90 (d, J = 2.1 Hz, 1H), 3.97 (t, J = 6.4 Hz, 2H), 3.66 (s, 3H), 2.31 (t, J = 7.4 Hz, 2H), 1.19 – 1.52 (m, 16H), 0.87 (t, J = 6.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 174.0 (s), 156.2 (s), 134.0 (d), 124.5 (d), 121.4 (s), 116.6 (d), 111.1 (s), 69.5 (t), 32.9 (t), 31.9 (t), 29.6 (t), 29.4 (t), 29.3 (t), 28.8 (t), 28.2 (t), 25.9 (t), 22.7 (t), 14.1 (q); IR (neat): ν = 2924, 2854, 1770, 1559, 1457, 1252, 1035 cm^{-1}; MS (EI) *m/z* 423 (M$^+$-Me), 343, 315, 280, 234, 219, 169, 135, 97, 85, 69, 43.

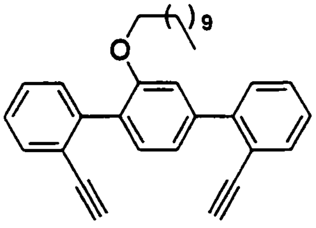
2,2''-Bis-trimethylsilanylethynyl-2'-undecyloxy-[1,1';4',1'']terphenyl



260

1-Bromo-2-(trimethylsilylethynyl)benzene (2.62 g, 10.3 mmol) in THF (75 mL) was cooled to -78°C and *n*-BuLi (6.2 mL, 1.66 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of $ZnBr_2$ (2.33 g, 10.3 mmol) in THF (75 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (450 mg, 0.39 mmol) and **258** (1.40 g, 3.44 mmol) in THF (30 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a yellow semi-solid. Alternatively, the reaction could be quenched by the addition of silica gel. Evaporating to dryness gives a crude yellow powder. Either crude product can be further purified by chromatography to give the product as a clear liquid (1.58 g, 78%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.56 (m, 2H), 7.37 (m, 3H), 7.18 – 7.42 (m, 6H), 3.96 (t, J = 6.5 Hz, 2H), 1.65 (m, 2H), 1.28 (m, 17H), 0.85 (t, J = 7.1 Hz, 3H), 0.14 (s, 9H), 0.05 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.7 (s), 144.4 (s), 141.9 (s), 140.9 (s), 133.5 (d), 132.1 (d), 130.7 (d), 130.3 (d), 129.3 (d), 129.1 (d), 128.7 (d), 127.9 (d), 126.8 (d), 126.6 (d), 123.1 (s), 121.4 (s), 120.8 (d), 113.3 (s), 105.1 (s), 105.0 (s), 97.6 (s), 96.4 (s), 68.4 (t), 65.8 (t), 31.9 (t), 29.6 (t), 29.6 (t), 29.5 (t), 29.4 (t), 29.3 (t), 29.2 (t), 26.0 (t), 22.7 (t), 15.2 (q), 14.1 (q), -0.14 (q), -0.17 (q); IR (neat): v = 2956, 2925, 2854, 2158, 1609, 1468, 1307, 1249, 1005, 758 cm^{-1}; MS (FAB) *m/z* 555, 505, 467, 420, 374, 331, 259, 215, 177, 149, 129, 97, 73, 45.

2,2''-Diethynyl-2'-undecyloxy-[1,1';4',1'']terphenyl

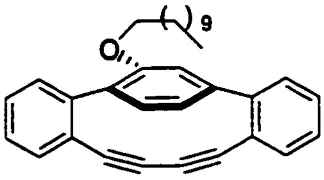


261

Compound **260** (1.5 g, 2.53 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a yellow gum (1.04 g, 99%). ^1H NMR (200 MHz, $CDCl_3$) δ 7.64 (m, 2H), 7.18 – 7.59 (m, 9H), 3.97 (t, J = 6.5 Hz, 2H), 3.10 (s, 1H), 2.95

(s, 1H), 1.68 (m, 2H), 1.23 (m, 16H), 0.89 (t, J = 6.6 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 155.7 (s), 144.2 (s), 141.5 (s), 140.8 (s), 134.0 (d), 133.0 (d), 130.9 (d), 130.6 (d), 129.6 (d), 129.0 (d, two overlapping signals), 128.2 (d), 127.0 (d), 126.7 (d), 122.0 (s), 120.8 (s), 120.3 (s), 113.5 (s), 83.3 (s, two overlapping signals), 80.3 (d), 79.3 (d), 68.4 (t), 31.9 (t), 29.6 (t, two overlapping signals), 29.5 (t), 29.3 (t), 29.2 (t), 29.1 (t), 26.0 (t), 22.7 (t), 14.1 (q); IR (neat): ν = 3297, 3097, 2934, 2852, 2101, 1607, 1550, 1463, 1206 cm^{-1}; MS (EI) *m/z* 448 (M^+), 348, 302, 276, 248, 194, 150, 57; HRMS Calcd for $C_{33}H_{36}O$ 448.2767 (M^+), found 448.2752.

1,3-Diyne-[4](2,2")-2'-undecyloxy-[1,1';4',1"]terphenylophane

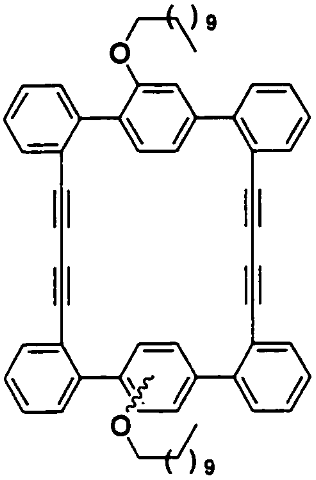


262

$Cu(OAc)_2$ (2.40 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (1000 mL) and **261** (1.04 mg, 2.20 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a yellow-black liquid (110 mg, 30%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (m, 2H), 7.45 (m, 2H), 7.30 (m, 4H), 7.148 (d, J = 7.5 Hz, 1H), 6.86 (m, 1H), 6.78 (s, 1H), 3.94 (m, 1H), 3.82 (m, 1H), 1.52 (m, 3H), 1.20 (m, 18H), 0.88 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.4 (s), 150.7 (s), 148.4 (s), 143.7 (s), 131.4 (d), 130.5 (s), 128.9 (s), 128.8 (d), 128.6 (d), 128.1 (d), 127.4 (s), 127.0 (d), 127.0 (d), 126.9 (d), 126.3 (d), 124.9 (s), 124.3 (s), 122.5 (d), 121.5 (s), 114.4 (d), 98.2 (s), 97.6 (s), 84.5 (s), 82.9 (s), 68.7 (t), 31.9 (t), 29.6 (t), 29.5 (t), 29.5 (t), 29.3 (t), 29.1 (t), 29.0 (t), 25.7 (t), 22.7 (t), 14.1 (q); IR (neat): ν = 3064, 2922, 2853, 2168, 1701, 1635, 1560, 1554, 1419, 1190, 754 cm^{-1}; MS (EI) *m/z*

446 (M^+), 404, 348, 333, 292, 263, 194, 165, 55, 43; HRMS Calcd for $C_{33}H_{34}O$ 446.2611, found 446.2615.

1,3,13,15-Tetrayne-[4.4](2,2")-2'-undecyloxy-[1,1';4',1"]terphenylophane

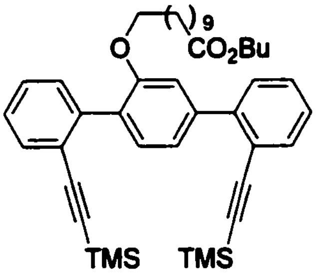


263

$Cu(OAc)_2$ (2.40 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (1000 mL) and **261** (1.04 g, 2.20 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a yellow gum (370 mg, 36%); ^1H NMR (500 MHz, $CDCl_3$) δ 7.8 (m, 4H), 7.20 – 7.44 (m, 16H), 7.13 (m, 2H), 3.96 (t, $J = 6.6$ Hz, 4H), 1.57 (m, 4H), 1.20 (m, 34H), 0.88 (t, $J = 6.9$ Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.7 (s), 155.8 (s), 155.8 (s), 144.8 (s), 144.7 (s), 142.4 (s), 142.1 (s), 140.7 (s), 140.5 (s), 134.2 (s), 134.1 (d), 133.1 (d), 133.0 (d), 130.8 (d), 130.6 (d), 130.4 (d), 130.4 (d), 129.3 (d), 129.2 (d), 129.1 (s), 129.0 (d), 128.5 (d), 128.3 (d), 127.0 (s), 126.9 (s), 126.8 (d), 126.7 (d), 122.1 (s), 122.0 (s), 121.0 (d), 120.9 (d), 120.4 (d), 120.2 (d), 113.8 (d), 113.7 (d), 81.4 (two overlapping signals, s), 80.8 (s), 80.7 (s), 77.0 (s), 76.9 (s), 75.9 (s), 75.7 (s), 69.2 (t), 69.1 (t), 31.9 (t), 29.6 (t), 29.6 (t), 29.5 (t), 29.5 (t), 29.3 (t), 29.3 (t), 29.2 (t), 29.1 (t), 29.1 (t), 25.9 (t), 25.8 (t), 22.7 (t), 14.1 (q); IR (neat): $\nu = 2923, 2852, 2319, 2163, 1694, 1571, 1419, 1210$ cm^{-1}; MS (FAB) *m/z* 893

(M^++H), 841, 767, 737, 676, 646, 567, 522, 491, 459, 430, 399, 338, 246, 137; Anal Calcd for $C_{66}H_{68}O_2$: C, 88.74; H, 7.68. Found: C, 89.01; H, 7.89.

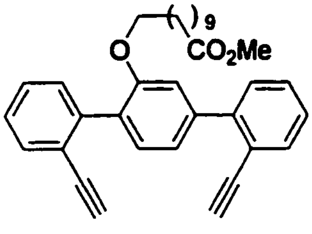
11-(2,2''-Bis-trimethylsilanylethynyl-[1,1';4',1'']terphenyl-2'-yloxy)-undecanoic acid butyl ester



264

1-Bromo-2-(trimethylsilylethynyl)benzene (2.40 g, 9.44 mmol) in THF (75 mL) was cooled to -78°C and *n*-BuLi (5.7 mL, 1.66 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of $ZnBr_2$ (2.12 g, 9.44 mmol) in THF (30 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (200 mg, 0.17 mmol) and **259** (1.40 g, 3.11 mmol) in THF (30 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a yellow semi-solid. Alternatively, the reaction could be quenched by the addition of silica gel. Evaporating to dryness gives a crude yellow powder. Either crude product can be further purified by chromatography to give the product as a clear liquid (1.077 g, 54%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (m, 2H), 7.37 (m, 5H), 7.24 (m, 4H), 4.08 (t, *J* = 6.5 Hz, 2H), 3.96 (t, *J* = 6.4 Hz, 2H), 2.35 (t, *J* = 6.2 Hz, 2H), 1.62 (m, 6H), 1.30 (m, 17H), 0.90 (t, *J* = 6.1 Hz, 3H), 0.32 (s, 9H), 0.23 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.7 (s), 155.6 (s), 144.2 (s), 141.8 (s), 140.9 (s), 133.4 (d), 132.0 (d), 130.6 (d), 130.2 (d), 129.2 (d), 129.0 (d), 128.6 (d), 127.8 (d), 126.7 (d), 126.5 (d), 123.0 (s), 121.3 (s), 120.7 (d), 113.2 (d), 105.1 (s), 104.9 (s), 97.4 (s), 96.2 (s), 68.3 (t), 63.9 (t), 34.2 (t), 30.6 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.2 (t), 29.1 (t), 29.0 (t), 25.9 (t), 24.9 (t), 19.0 (t), 15.1 (q), 13.6 (q), -0.24 (q), -0.33 (q); IR (neat): ν = 2829, 2855, 2157, 1652, 1571, 1516, 1405, 1307, 1249, 759 cm^{-1}; MS (FAB) *m/z* 679 (M^++H), 591, 407, 335, 241, 147, 73.

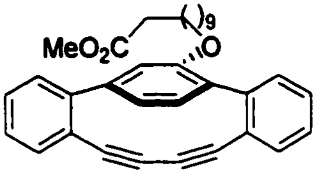
11-(2,2''-Diethynyl-[1,1';4',1'']terphenyl-2'-yloxy)-undecanoic acid methyl ester



265

Compound **264** (1.04 g, 1.53 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a yellow liquid (611 mg, 75%). ^1H NMR (200 MHz, $CDCl_3$) δ 7.64 (m, 2H), 7.19 – 7.60 (m, 9H), 3.98 (t, J = 6.4 Hz, 2H), 3.66 (s, 3H), 3.11 (s, 1H), 2.31 (t, J = 7.4 Hz, 2H), 1.64 (m, 2H), 1.24 (m, 16H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 174.0 (s), 155.7 (s), 144.2 (s), 141.5 (s), 140.8 (s), 134.0 (d), 132.8 (d), 130.8 (d), 130.5 (d), 129.5 (d), 129.0 (s), 128.9 (d), 128.0 (d), 126.9 (d), 126.7 (d), 122.0 (s), 120.8 (s), 120.3 (s), 113.6 (d), 83.3 (s, two overlapping signals), 80.3 (d), 79.3 (d), 68.4 (t), 51.2 (q), 34.0 (t), 29.3 (t), 29.2 (t), 29.1 (t, overlapping signals), 29.0 (t, overlapping signals), 25.9 (t), 24.9 (t); IR (neat): ν = 3286, 2929, 2858, 2098, 1736, 1603, 1467, 1203 cm^{-1}; MS (EI) *m/z* 492 (M^+), 461, 419, 294, 265, 194, 162, 112, 31; HRMS Calcd for $C_{34}H_{36}O_3$ 492.2666 (M^+), found 492.2671.

1,3-Diyne-[4](2,2'')-[(11-carboxymethyl)-2'-undecyloxy-[1,1';4',1'']terphenylophane

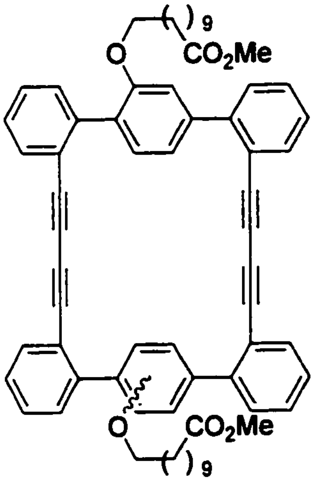


266

$Cu(OAc)_2$ (1.32 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (400 mL) and **265** (600 mg, 1.21 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump.

The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a reddish-black liquid (268 mg, 45%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (m, 2H), 7.31 (m, 7H), 6.86 (d, J = 7.6 Hz, 1H), 6.78 (s, 1H), 3.93 (m, 1H), 3.82 (m, 1H), 3.65 (s, 3H), 2.30 (t, J = 7.5 Hz, 2H), 1.53 (m, 4H), 1.22 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.2 (s), 158.3 (s), 150.7 (s), 148.4 (s), 143.6 (s), 131.4 (d), 130.4 (d), 128.8 (d), 128.6 (d), 128.1 (d), 127.4 (d), 127.0 (d), 126.9 (d), 126.9 (d), 126.2 (d), 124.9 (s), 124.3 (d), 122.5 (d), 114.4 (d), 98.2 (s), 97.6 (s), 84.6 (s), 83.0 (s), 51.3 (t), 34.1 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.1 (t), 29.0 (t), 28.9 (t), 25.7 (t), 24.9 (t); IR (neat): v = 2926, 2853, 2163, 1711, 1684, 1652, 1203 cm^{-1}; MS (EI) *m/z* 490 (M^+), 459, 443, 387, 323, 292, 263, 217, 155, 119, 83, 55, 41; HRMS Calcd for $C_{34}H_{34}O_3$ 490.2509, found 490.2496.

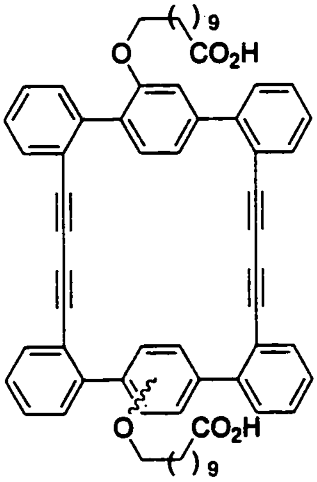
1,3,13,15-Tetrayne-[4.4](2,2")-[(11-carboxymethyl)-2'-undecyloxy-[1,1';4',1"]terphenylophane



267

$Cu(OAc)_2$ (1.32 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (400 mL) and **265** (600 mg, 1.21 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a bright yellow film (103 mg, 17%); ^1H NMR (500 MHz, $CDCl_3$) δ 8.61 (m, 2H), 7.52 – 7.66 (m, 4H), 7.12 – 7.49 (m, 8H), 3.94 (m, 4H), 3.64 (s, 6H), 2.28 (t, J = 7.2 Hz, 4H), 1.56 (m, 8H), 1.20 (m, 28H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.2 (s), 155.9 (s), 155.7 (s), 149.7 (s), 144.7 (s), 144.6 (s), 142.4 (s), 142.1 (s), 140.7 (s), 140.5 (s), 140.4 (s), 134.2 (d), 134.1 (d), 133.1 (d), 133.0 (d), 130.8 (d), 130.6 (d), 130.4 (d), 130.3 (d), 129.5 (d), 129.3 (d), 129.1 (d), 129.1 (d), 128.5 (d), 128.3 (d), 127.0 (s), 126.9 (s), 126.8 (d), 126.7 (d), 122.1 (d), 122.0 (d), 121.0 (d), 120.9 (d), 120.3 (d), 120.1 (d), 113.8 (d), 113.7 (d), 81.4 (two overlapping signals, s), 80.8 (s), 80.7 (s), 76.9 (two overlapping signals, s), 75.9 (s), 75.6 (s), 69.1 (t), 69.0 (t), 51.4 (q), 34.1 (t), 30.3 (t), 29.6 (t), 29.4 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.2 (t), 29.1 (t), 29.1 (t), 29.0 (t), 26.0 (t), 25.9 (t), 25.8 (t), 24.9 (t), 24.9 (t); IR (neat): ν = 2929, 2853, 2319, 1735, 1570, 1437, 1209 cm^{-1}; MS (FAB) *m/z* 645, 553, 461, 369, 277, 241, 185, 132, 93.

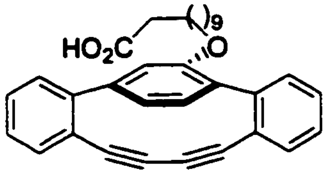
1,3,13,15-Tetrayne-[4.4](2,2")-[(11-carboxy)-2'-undecyloxy-[1,1';4',1"]terphenylophane



269

Excess $LiOH•H_2O$ was added to a solution of **267** (100 mg, 0.10 mmol) in THF (15 mL), MeOH (2 mL), H_2O (2 mL) and stirred for 1 hr. The solution was separated between Et_2O and H_2O and the organic phase washed with 10% HCl and brine, dried and isolated as a dull yellow solid (95 mg, 98%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.61 (m, 4H), 7.20 – 7.44 (m, 14H), 7.14 (m, 4H), 3.99 (t, *J* = 6.4 Hz, 4H), 2.33 (t, *J* = 7.3 Hz, 4H), 1.60 (m, 8H), 1.21 (m, 36H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 179.9 (s), 165.7 (s), 155.8 (s), 155.8 (s), 144.8 (s), 144.6 (s), 142.4 (s), 142.1 (s), 140.7 (s), 140.5 (s), 134.2 (d), 134.1 (d), 133.1 (d), 133.0 (d), 130.8 (d), 130.6 (d), 130.4 (d), 130.4 (d), 129.3 (d), 129.2 (d), 129.1 (d), 129.1 (d), 128.5 (d), 128.3 (d), 127.0 (s), 126.9 (s), 126.8 (d), 126.7 (d), 122.1 (d), 122.0 (d), 121.0 (d), 120.9 (d), 120.3 (d), 120.1 (d), 113.8 (d), 113.7 (d), 81.4 (two overlapping signals, s), 80.8 (s), 80.7 (s), 77.0 (s), 76.9 (s), 75.9 (s), 75.7 (s), 69.1 (t), 69.0 (t), 34.0 (t), 29.6 (t), 29.4 (t), 29.2 (t), 29.2 (t), 29.1 (t), 29.1 (t), 29.1 (t), 29.0 (t), 28.9 (t), 25.8 (t), 25.8 (t), 24.6 (t), 24.5 (t); IR (neat): *ν* = 3302, 2924, 2858, 2319, 1734, 1635, 1560, 1209 cm^{-1}; MS (FAB) *m/z* 953 (M^++H), 842, 768, 537, 391, 219, 149, 57; Anal Calcd for $C_{66}H_{66}O_6$: C, 83.15; H, 6.77. Found: C, 83.01; H, 6.66.

1,3-Diyne-[4](2,2")-[(11-carboxy)-2'-undecyloxy-[1,1';4',1"]terphenylophane

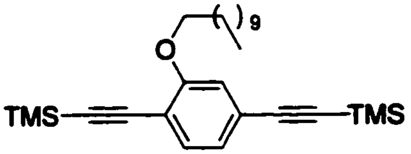


268

Excess LiOH•H_2O was added to a solution of **266** (150 mg, 0.31 mmol) in THF (10 mL), MeOH (5 mL), H_2O (5 mL) and stirred for 1 hr. The solution was separated between Et_2O and H_2O and the organic phase washed with 10% HCl and brine, dried and isolated as a brown semi-solid (105 mg, 72%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.64 (dd, *J* = 6.4, 0.6 Hz, 1H), 7.43 (dt, *J* = 7.5, 1.3 Hz, 2H), 7.28 (m, 2H), 7.23 (m, 2H), 7.11 (d, *J* = 7.6 Hz, 1H), 6.86 (dd, *J* = 7.6, 1.5 Hz, 1H), 6.77 (d, *J* = 1.5 Hz, 2H), 3.95 (m, 1H), 3.80 (m, 1H), 2.33 (t, *J* = 7.5 Hz, 1H), 1.61 (m, 4H), 1.22 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 179.6 (s), 158.3 (s), 150.6 (s), 143.6 (s), 131.4 (d), 130.4 (s), 128.8 (d), 128.6 (d), 128.0 (d), 127.3 (d), 127.0 (s), 126.9 (d), 126.8 (d), 126.2 (d), 124.8 (s), 124.2 (s), 122.4 (d), 114.4 (d), 98.2 (s), 97.6 (s), 84.4 (s), 82.9 (s), 68.6 (t), 33.9 (t), 29.4 (t), 29.2 (t), 29.1 (t), 29.0 (t), 29.0 (t), 28.9 (t), 25.6 (t), 24.6 (t); IR (neat): *ν* =

2926, 2853, 2163, 1706, 1684, 1652, 1203 cm^{-1}; MS (EI) *m/z* 476 (M^+), 419, 332, 292, 263, 205, 162, 98, 55, 28.

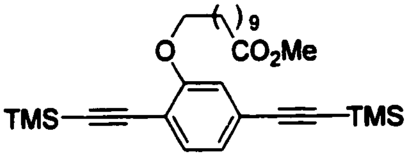
1,4-Bis-trimethylsilanylethynyl-2-undecyloxybenzene



270

CuI (60 mg, 0.32 mmol), $Pd(PPh_3)_4$ (120 mg, 0.17 mmol) and **258** (2.60 g, 6.40 mmol) were added to a flame dried round bottom flask and dissolved in DEA (40 mL) and THF (40 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar or N_2. Trimethylsilylacetylene (1.9 mL, 13.5 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide a slightly pale yellow liquid (2.20 g, 79%); ^1H NMR (200 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.9 Hz, 1H), 6.93 (dd, *J* = 7.9, 1.3 Hz, 1H), 6.87 (d, *J* = 1.3 Hz, 1H), 3.93 (t, *J* = 6.0 Hz, 2H), 1.80 (m, 2H), 1.50 (m, 2H), 1.20 – 1.38 (m, 13H), 0.87 (t, *J* = 1.7 Hz, 3H), 0.23 (s, 9H), 0.22 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 159.7 (s), 133.1 (d), 124.2 (s), 123.9 (d), 114.7 (d), 113.2 (s), 104.8 (s), 100.9 (s), 99.9 (s), 95.4 (s), 68.4 (t), 31.9 (t), 29.6 (t), 29.3 (t), 29.3 (t), 29.1 (t), 25.9 (t), 22.6 (t), 14.0 (q), -0.12 (q), -0.18 (q); IR (neat): *v* = 2957, 2924, 2855, 2159, 1598, 1499, 1249, 861 cm^{-1}; MS (EI) *m/z* 440 (M^+), 370.2, 271, 226, 162, 73, 43; HRMS Calcd for $C_{27}H_{44}OSi_2$ 440.2932 (M^+), found 440.2940.

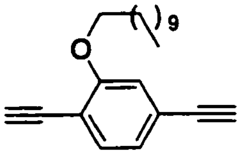
11-(2,5-Bis-trimethylsilanylethynyl-phenoxy)-undecanoic acid methyl ester



271

CuI (30 mg, 0.16 mmol), $Pd(PPh_3)_4$ (50 mg, 0.07 mmol) and **259** (1.30 g, 2.88 mmol) were added to a flame dried round bottom flask and dissolved in DEA (15 mL) and THF (15 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar. Trimethylsilylacetylene (0.860 mL, 5.77 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide a slightly pale yellow liquid (1.21 g, 87%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.8 Hz, 1H), 6.94 (dd, *J* = 7.8, 1.2 Hz, 1H), 6.88 (s, 1H), 3.96 (t, *J* = 6.1 Hz, 2H), 3.64 (s, 3H), 2.27 (t, J= 7.3 Hz, 2H), 1.85 (m, 2H), 1.19 – 1.61 (m, 13H), 0.82 (m, 2H), 0.22 (s, 18H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 174.2 (s), 159.8 (s), 133.3 (d), 124.2 (s), 124.1 (d), 115.1 (d), 113.4 (s), 104.8 (s), 101.0 (s), 100.2 (s), 98.1 (s), 68.7 (t), 51.3 (q), 34.1 (t), 29.5 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.1 (t), 26.0 (t), 25.0 (t), 22.6 (t), -0.04 (q), -0.10 (q); IR (neat): *ν* = 2955, 2928, 2855, 2185, 1742, 1598, 1249 cm^{-1}; MS (EI) *m/z* 484 (M^+), 419, 313, 271, 149, 73, 55; HRMS Calcd for $C_{28}H_{44}O_3Si_2$ 484.2830 (M^+), found 484.2853.

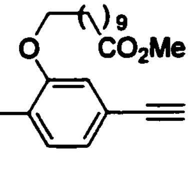
1,4-Bisethynyl-2-undecyloxybenzene



272

Compound **270** (2.40 g, 5.45 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a pale yellow liquid (1.31 g, 81%). ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.7 Hz, 1H), 7.00 (dd, *J* = 7.7, 1.4 Hz, 1H), 6.95 (d, *J* = 1.4 Hz, 1H), 3.99 (t, *J* = 6.6 Hz, 2H), 3.31 (s, 1H), 3.13 (s, 1H), 1.81 (m, 2H), 1.19 – 1.45 (m, 16H), 0.86 (t, *J* = 6.1 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 159.9 (s), 133.8 (d), 124.1 (d), 123.5 (s), 115.3 (d), 112.7 (s), 83.2 (s), 82.5 (d), 79.5 (s), 78.5 (d), 68.9 (t), 31.9 (t), 29.5 (t), 29.3 (t), 29.3 (t), 28.9 (t), 25.8 (t), 22.6 (t), 14.0 (q); IR (neat): *ν* = 3298, 2924, 2854, 2107, 1600, 1550, 1273, 1117 cm^{-1}; MS (EI) *m/z* 296 (M^+), 267, 239, 211, 185, 169, 142, 114, 83, 69, 55, 43, 29; HRMS Calcd for $C_{21}H_{28}O$ 296.2141 (M^+), found 296.1777.

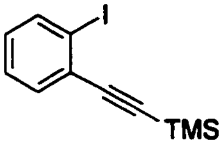
11-(2,5-Bis-ethynyl-phenoxy)-undecanoic acid methyl ester



273

Compound **271** (580 m g, 1.19 mmol) dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography (Pet Ether) yielded the product as a yellow solid (350 mg, 82%). ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.36 (d, J = 7.7 Hz, 1H), 7.00 (dd, J = 7.7, 1.3 Hz, 1H), 6.95 (d, J = 1.3 Hz, 1H), 4.00 (t, J = 6.5 Hz, 2H), 3.64 (s, 3H), 3.31 (s, 1H), 3.13 (s, 1H), 2.28 (t, J = 8.1 Hz, 2H), 1.81 (m, 2H), 1.19 – 1.59 (m, 16H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 174.2 (s), 159.9 (s), 133.9 (d), 124.2 (d), 123.6 (s), 115.4 (d), 112.7 (s), 83.3 (s), 82.6 (d), 79.6 (s), 78.5 (d), 68.9 (t), 51.4 (s), 34.1 (t), 29.6 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.1 (t), 29.0 (t), 25.9 (t), 25.0 (t); IR (neat): v = 3288, 2924, 2857, 2101, 1744, 1595, 1548, 1263 cm^{-1}; MS (EI) m/z 340 (M^+), 283, 230, 169, 142, 114, 97, 83, 69, 55; HRMS Calcd for $C_{22}H_{28}O_3$ 340.2039 (M^+), found 340.2034.

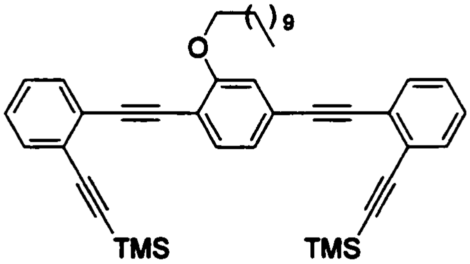
1-Iodo-2-(trimethylsilylethynyl)benzene[31,32,33]



274

n-BuLi (5.8 mL, 13.8 mmol) was added to a solution of 1-bromo-2-(trimethylsilylethynyl)benzene (3.5 g, 13.8 mmol) in THF (130 mL) at -78°C and the resulting yellow solution stirred for 15 minutes. A solution of NIS (3.13 g, 13.8 mmol) in THF (20 mL) at -78°C was transferred via canula to the solution of the aryllithium. The cold bath was removed and the solution was allowed to come to RT and stir overnight. The solution was poured into ether and washed with water and brine, dried and evaporated to yield a dark yellow oil. The oil was purified by chromatography (Pet. Ether) to provide the product as a slightly pale yellow liquid. (3.7 g, 90%); ^{1}H NMR (500 MHz, CDCl$_3$) 7.82 (dd, J = 7.6, 1.9 Hz, 1H), 7.45 (dd, J = 7.0, 1.1 Hz, 1H), 7.27 (m, 1H), 6.95 (dt, J = 7.0, 1.0 Hz, 1H), 0.24 (s, 9H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 138.6 (d), 132.7 (d), 131.9 (s), 129.5 (d), 128.1 (s), 127.6 (d), 106.5 (s), 98.7 (s), -0.19 (q); IR (neat): ν = 3065, 2960, 2899, 2351, 1585, 1465, 1429, 1251, 1120, 1027, 945, 757 cm^{-1}; MS (EI) m/z 360 (M$^+$), 143, 131, 115, 79, 69, 43; HRMS Calcd for $C_{11}H_{13}ISi$ 360.0910, found 360.0922; Anal Calcd for $C_{11}H_{13}ISi$: C, 44.01; H, 4.36. Found: C, 43.74; H,.4.36.

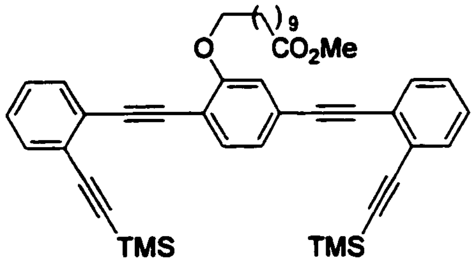
1,4-((*o*-Trimethylsilylethynylphenyl)ethynyl)-2-undecyloxybenzene



275

CuI (60 mg, 0.32 mmol), Pd(PPh$_3$)$_4$ (60 mg, 0.09 mmol), and **186** (300 mg, 4.33 mmol) were added to a flame dried round bottom flask and dissolved in DEA (50 mL) and THF (50 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar or N$_2$. Compound **272** (610 g, 2.06 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide the product as a thick red oil (1.06 g, 40%). ^{1}H NMR (200 MHz, CDCl$_3$) δ 7.52 (m, 5H), 7.00 – 7.39 (m, 6H), 4.05 (t, J = 6.4 Hz, 2H), 1.87 (m, 2H), 1.34 (m, 17H), 0.88 (t, J = 5.8 Hz, 3H), 0.30 (s, 9H), 0.28 (s, 9H); ^{13}C NMR (50 MHz, CDCl$_3$) δ 159.3 (s), 133.3 (d), 132.2 (d), 132.1 (d), 131.6 (d), 128.2 (d), 128.0 (d), 127.7 (d), 126.4 (s), 125.8 (s), 125.7 (s), 125.5 (s), 124.3 (s), 124.1 (d), 114.9 (d), 113.6 (s), 103.6 (s), 103.5 (s), 98.6 (s), 98.4 (s), 93.8 (s), 93.3 (s), 89.9 (s), 89.6 (s), 68.9 (t), 65.7 (t), 53.3 (t), 31.8 (t), 29.6 (t), 29.4 (t), 29.3 (t), 29.2 (t), 26.0 (t), 25.9 (t), 22.6 (t), 14.0 (q), -0.00 (q), -0.06 (q); IR (neat): ν = 2956, 2854, 2159, 1598, 1543, 1413, 1234, 1094, 869 cm^{-1}; MS (FAB) m/z 640, 567, 524, 499, 458, 355, 309, 259, 203, 149, 97, 75, 57, 40.

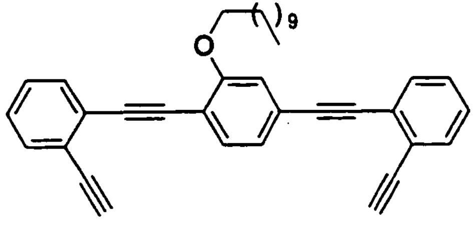
1,4-((*o*-Trimethylsilylethynylphenyl)ethynyl)-[(11'-carboxymethyl)-2-undecyloxy]-benzene



276

CuI (20 mg, 0.01 mmol), $Pd(PPh_3)_4$ (30 mg, 0.004 mmol), and **186** (680 mg, 2.26 mmol) were added to a flame dried round bottom flask and dissolved in DEA (15 mL) and THF (15 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar or N_2. Compound **273** (350 mg, 1.03 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide the product as a thick yellow oil (285 g, 61%). ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.49 (m, 5H), 7.27 – 7.05 (m, 6H), 4.04 (t, *J* = 6.4 Hz, 2H), 3.63 (s, 3H), 2.26 (t, *J* = 6.5 Hz, 2H), 1.89 (m, 2H), 1.40 (m, 16H), 0.86 (m, 3H), 0.27 (s, 9H), 0.25 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 174.1 (s), 159.3 (s), 133.3 (d), 132.2 (d), 132.2 (d), 131.7 (d), 128.2 (d), 128.1 (d), 127.7 (d), 126.4 (s), 125.8 (s), 125.7 (s), 125.5 (s), 124.4 (s), 124.1 (d), 115.0 (d), 113.7 (s), 103.6 (s), 103.5 (s), 98.7 (s), 98.5 (s), 93.8 (s), 93.3 (s), 89.9 (s), 89.6 (s), 69.0 (t), 51.3 (t), 34.0 (t), 29.5 (t), 29.3 (t), 29.2 (t), 29.1 (t), 26.0 (t), 24.9 (t), 0.02 (q), -0.04 (q); IR (neat): v = 2927, 2854, 2159, 1738, 1543, 1249, 759 cm^{-1}; MS (FAB) *m/z* 684 (M^+), 553, 508, 418, 354, 218, 138, 72.

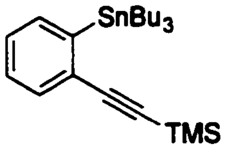
1,4-((*o*-ethynylphenyl)ethynyl)-2-undecyloxybenzene



277

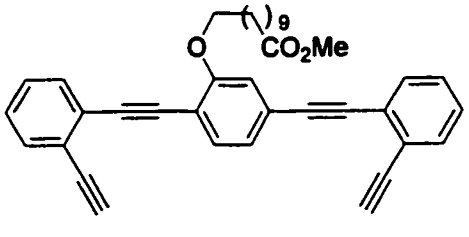
Compound **275** (1.02 g, 2.11 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and K_2CO_3 (excess) was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography (Pet Ether) yielded the product as a deep yellow gum (780 mg, 95%). ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.47 – 7.57 (m, 5H), 7.24 – 7.30 (m, 4H), 7.14 (d, J = 7.9 1H), 7.06 (s, 1H), 4.05 (t, J = 6.2 Hz, 2H), 3.72 (m, 2H), 3.39 (s, 1H), 3.35 (s, 1H), 1.78 – 1.88 (m, 3H), 1.24 – 1.52 (m, 12H), 0.87 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 160.0 (s), 134.1 (d), 133.3 (d), 132.6 (d), 132.4 (d), 129.2 (d), 129.1 (d), 128.8 (d), 128.5 (d), 127.2 (d), 126.6 (s), 125.3 (s), 125.0 (s), 124.7 (s), 124.0 (d), 115.5 (d), 114.0 (s), 94.1 (s), 90.6 (s), 90.0 (s), 82.9 (s), 82.8 (s), 82.0 (d), 81.8 (d), 69.5 (t), 68.6 (t), 32.6 (t), 30.3 (t), 30.1 (t), 30.0 (t), 29.9 (t), 26.7 (t), 23.4 (t), 14.8 (q); IR (neat): ν = 3290, 2922, 2853, 2365, 1685, 1654, 1508 cm^{-1}; MS (EI) *m/z* 496 (M^+), 450, 419, 386, 332, 296, 262, 213, 162, 112, 51; HRMS Calcd for $C_{37}H_{36}O$ 496.2767 (M^+), found 496.2757.

1-Tri-*n*-butyltin-2-(trimethylsilylethynyl)benzene



n-BuLi (430 uL, 1.04 mmol) was added to a solution of 1-bromo-2-(trimethylsilylethynyl)benzene (264 mg, 1.04 mmol) in THF (10 mL) at -78°C and the resulting yellow solution stirred for 15 minutes. To this solution was added tri-*n*-butyltinchloride (321.4 mg, 1.00 mmol) the solution was allowed to come to RT and stir overnight. The solution was poured into ether and washed with water and brine, dried and evaporated to yield a slightly yellow oil. The oil was purified by chromatography (Pet. Ether) to provide the product as a clear liquid. (450 mg, 90%); ^{1}H NMR (500 MHz, $CDCl_3$) 7.49 (m, 2H), 7.25 (m, 2H), 1.60 (m, 6H), 1.33 (m, 6H), 1.20 (m, 6H), 0.92 (t, J = 7.3 Hz, 9H), 0.29 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) 146.0 (s), 136.1 (d), 132.4 (d), 130.7 (s), 127.8 (d), 127.6 (d), 107.8 (s), 93.6 (s), 29.6 (t), 27.4 (t), 13.7 (q), 9.9 (t), -0.08 (q); IR (neat); ν = 2957, 2924, 2362, 2152, 1457, 1429, 1250, 1073, 864, 842, 759 cm^{-1}; MS (EI) *m/z* 407 (M^+-Bu), 351, 295, 220, 159, 73, 41; Anal Calcd for $C_{23}H_{40}SnSi$: C, 59.63; H, 8.70. Found: C, 59.80; H,.8.86

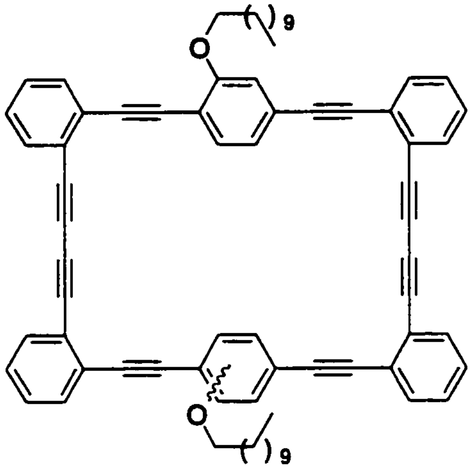
1,4-((*o*-ethynylphenyl)ethynyl)-[(11'-carboxymethyl)-2-undecyloxy]-benzene



278

Compound **276** (420 mg, 0.61 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and K_2CO_3 (excess) was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as an orange gum (203 mg, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (m, 3H), 7.50 (d, *J* = 1.3 Hz, 2H), 7.29 (m, 2H), 7.12 (dd, *J* = 6.5, 1.4 Hz, 2H), 7.04 (d, *J* = 1.4 Hz, 2H), 4.05 (t, *J* = 6.5 Hz, 2H), 3.63 (s, 3H), 3.37 (s, 1H), 3.33 (s, 1H), 2.26 (t, J = 7.4 Hz, 2H), 1.82 (m, 2H), 1.55 (m, 5H), 1.27 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.2 (s), 159.3 (s), 133.4 (d), 133.1 (s), 132.6 (d), 132.6 (d), 132.0 (d), 131.8 (d), 128.5 (d), 128.4 (d), 128.1 (d), 127.8 (s), 126.6 (s), 126.0 (s), 124.6 (s), 124.3 (s), 124.3 (s), 124.0 (d), 115.0 (d), 113.5 (s), 93.4 (s), 90.0 (s), 89.3 (s), 82.2 (s), 82.1 (s), 81.2 (d), 81.1 (d), 68.8 (t), 51.3 (q), 34.0 (t), 29.5 (t), 29.3 (t), 29.2 (t), 29.2 (t), 29.1 (t), 26.0 (t), 24.9 (t), 14.8 (q); IR (neat): *v* = 2929. 2852, 2139, 1744, 1652, 1564, 1416 cm^{-1}; MS (FAB) *m/z* 541 (M^++H), 342, 313, 277, 215, 186, 133, 77.

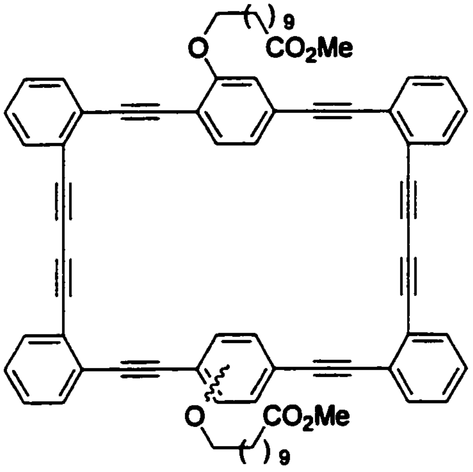
3,9-Di-*o*-phenyl-1,5,7,11-tetrayne-[12.12]-2-undecyloxyparacyclophane



279

$Cu(OAc)_2$ (1.60 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (1000 mL) and **277** (740 mg, 1.48 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids could be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a bright yellow solid (198 mg, 13%); ^1H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.55 (m, 4H), 7.22 – 7.33 (m, 5H), 6.81 – 6.84 (m, 2H), 3.93 (t, J = 6.5 Hz, 2H), 1.63 (m, 2H), 1.00 – 1.27 (m, 18H), 0.87 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.9 (s), 158.7 (s), 133.9 (d), 133.4 (d), 132.1 (d), 132.06 (d), 131.9 (d), 131.8 (d), 131.4 (d), 131.3 (d), 131.1 (d), 131.0 (d), 129.0 (s), 128.9 (s), 128.8 (s), 128.7 (s), 128.4 (s), 128.2 (s), 128.0 (d), 127.9 (d), 127.7 (d), 127.6 (d), 127.6 (d), 127.5 (d), 125.0 (s), 124.7 (s), 124.6 (s), 124.3 (s), 123.8 (d), 123.4 (d), 114.5 (d), 114.2 (d), 112.9 (s), 112.5 (s), 95.4 (s), 95.3 (s), 92.8 (s), 92.6 (s), 92.0 (s), 91.7 (s), 88.8 (s), 88.7 (s), 88.6 (s), 81.5 (s), 81.4 (s), 81.3 (s), 81.2 (s), 78.4 (s), 78.2 (s), 78.1 (s), 77.9 (s), 68.6 (t), 68.5 (t), 31.9 (t), 30.3 (t), 29.6 (t), 29.6 (t), 29.5 (t), 29.5 (t), 29.3 (t), 29.3 (t), 29.1 (t), 29.0 (t), 25.8 (t), 25.7 (t), 22.9 (t), 22.7 (t), 14.1 (q), 14.0 (q); IR (neat): ν = 2924, 2853, 2209, 1600, 1544, 1228 cm^{-1}; MS (FAB) *m/z* 989 (M^++H), 836, 735, 644, 582, 490, 458, 403, 337, 305.

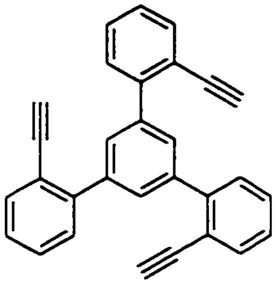
3,9-Di-*o*-phenyl-1,5,7,11-tetrayne-[12.12]-[(11'-carboxymethyl)-2-undecyloxy]-paracyclophane



280

$Cu(OAc)_2$ (370 mg, 6 equiv.) was dissolved in pyridine: ether (3:1) (160 mL) and **278** (184 mg, 0.34 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids could be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as a brown gum (30 mg, 17%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (m, 8H), 7.30 (m, 12H), 6.85 (m, 4H), 3.92 (t, *J* = 6.5 Hz, 4H), 3.65 (s, 6H), 2.32 (m, 5H), 1.63 (m, 14H), 1.20 (m, 34H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.2 (s), 158.9 (s), 158.7 (s), 133.9 (d), 133.4 (d), 132.1 (d), 132.1 (d), 131.9 (d), 131.4 (d), 131.3 (d), 131.1 (d), 130.0 (d), 129.0 (s), 129.0 (s), 128.7 (s), 128.3 (s), 128.2 (s), 128.0 (s), 127.9 (d), 127.7 (d), 127.6 (d), 127.6 (d), 127.5 (d), 125.0 (d), 124.7 (s), 124.6 (s), 124.3 (s), 123.8 (d), 123.4 (d), 123.4 (d), 114.4 (d), 114.3 (d), 112.9 (s), 112.5 (s), 95.4 (s), 95.3 (s), 92.8 (s), 92.6 (s), 92.0 (s), 91.7 (s), 88.8 (s), 88.6 (s), 81.5 (s), 81.4 (s), 81.3 (s), 81.2 (s), 78.4 (s), 78.2 (s), 78.1 (s), 78.0 (s), 68.6 (t), 68.5 (t), 51.4 (q), 34.1 (t), 29.6 (t), 29.5 (t), 29.4 (t), 29.4 (t), 29.4 (t), 29.3 (t), 29.1 (t), 29.1 (t), 29.0 (t), 25.7 (t), 25.6 (t), 24.9 (t); IR (neat): ν = 2910, 2852, 2202, 1704, 1696, 1635, 1437, 668 cm^{-1}; MS (FAB) *m/z* 1077 (M^++H), 921, 798, 738, 707, 646, 615, 554, 523, 430, 338, 241, 186, 137, 94; Anal Calcd for $C_{76}H_{68}O_6$: C, 84.72; H, 6.37. Found: C, 84.55; H, 6.37.

1,3,5-(*o*-Ethynylphenyl)benzene



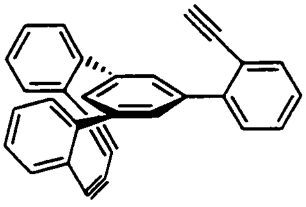
281

From **286a**: Compound **286a** (507 mg, 0.60 mmol) was dissolved in THF (30 mL) and TBAF (2.0 mL, 1.0 M in THF) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was further purified by chromatography to give the product as a yellow solid (157 mg, 70%).

From **286b**: Compound **286b** (1.5 g, 2.52 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a white solid (820 mg, 86%) m.p. 151-152°C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.84 (s, 3H), 7.63 (m, 3H), 7.49 (m, 3H), 7.33 (dt, J = 7.7, 1.2 Hz, 3H), 7.29 (dt, J = 7.6, 1.3 Hz, 3H), 3.08 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.2 (s), 139.6 (s), 133.9 (d), 129.8 (d), 129.3 (d), 129.0 (d), 127.1 (d), 120.5 (s), 83.3 (s), 80.4 (d); IR ($CHCl_3$): ν = 3059, 3028, 2107, 1484, 1447, 1412, 1226, 1161, 1113, 1039, 951, 893.5, 873, 758 cm^{-1}; MS (FAB) *m/z* (relative intensity) 379 (M^++H), 363, 289, 239, 189, 154, 133, 115, 89, 77, 63.

Crystal size 0.15 x 0.15 x 0.04 mm^3, triclinic, space group *P-1*, scan range 1.56<θ<20.81°, a=11.650(2), b=14.144(3), c=15.046(3) Å, ß=68.153°, V =2125.3 $Å^3$, Z=4, ρ_{calcd}=1.183 g cm-3, u=0.067 mm^{-1}, 4427 unique reflections at -80°C, of which 16532 were taken as observed [I_o>2.00σ(I)], R=0.0599, R_w=0.1069.

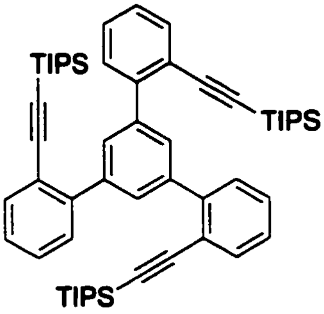
1,3-Diyne-[4](2,2")[1,1';3',1"]-(5'-*o*-ethynylphenyl)terphenylophane



283

$Cu(OAc)_2$ (2.00 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (320 mL) and 1,4-(*o*-ethynylphenyl)benzene (192 mg, 0.69 mmol) in 20 mL of pyridine:ether (3:1) was added over 3 hr *via* syrringe pump. The solution was either protected from light with Aluminum foil or left uncovered, and stirred at RT for 3 days. The solution was then poured into ether and 1 M HCL. The organic phase was washed with 1 M HCl until all pyridine was removed, saturated $NaHCO_3$ (aq) and brine, dried and evaporated to yield a yellow solid. The solid was further purified by chromatography (6:1 Hexanes: CH_2Cl_2) to yield the product as a white solid that reddened to an oily semisolid when left standing; ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.38 (t, *J* = 1.7 Hz, 1H), 7.76 (m,2H), 7.64 (m, 2H), 7.64 (d, *J* = 1.7 Hz, 2H), 7.55 (dt, *J* = 7.2, 1.4 Hz, 2H), 7.44 (m, 2H), 7.40 (m, 2H), 7.34 (dd, *J*= 7.7, 1.2 Hz, 2H), 2.80 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.4 (s), 144.3 (s), 140.4 (s), 140.3 (s), 137.3 (d), 134.8 (d), 131.8 (d), 131.4 (d), 131.3 (d), 130.5 (d), 130.2 (d), 128.8 (d), 128.4 (d), 128.3 (d), 122.1 (s), 121.4 (s), 108.9 (d), 87.4 (s), 84.1 (s), 82.6 (s); IR (film): *v* = 3288, 3059, 2955, 2925, 2171, 1725, 1560, 1473, 1216, 1110, 894, 757 cm^{-1}; MS (EI) *m/z* 376 (M^+), 255, 219, 137, 69; HRMS calcd for $C_{30}H_{16}$ 376.1252, found 376.1261.

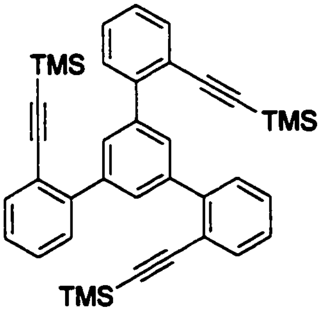
1,3,5-(*o*-Triispropylsilylethynylphenyl)benzene



286a

1-Bromo-2-(triisopropylsilylethynyl)benzene, **185** (999 mg, 2.97 mmol) in THF (30 mL) was cooled to -78°C and *n*-BuLi (1.17 mL, 2.97 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (667 mg, 2.97 mmol) in THF (30 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (90 mg, 0.077 mmol) and 1,3,5-tribromobenzene (208 mg, 0.66 mmol) in THF (20 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction could be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography (Pet Ether) to give the product as a pale yellow solid (437 mg, 78%). ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.12 (s, 3H), 7.38 (m, 12H), 0.89 (s, 63H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 143.2 (s), 139.6 (s), 134.4 (d), 129.9 (d), 129.3 (d), 128.4 (d), 126.7 (d), 121.8 (s), 106.5 (s), 94.4 (s), 18.5 (q), 11.2 (d); IR ($CHCl_3$): ν = 3021, 2947, 2865, 2153, 1464, 1229, 995, 883 cm^{-1}; MS (FAB) *m/z* 847 (M^++H), 553, 399, 307, 246, 137, 93; Anal Calcd for $C_{57}H_{78}Si_3$: C, 80.76; H, 9.29. Found: C, 81.00; H, 9.12.

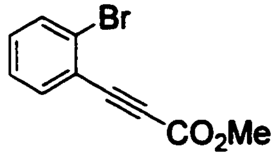
1,3,5-(*o*-Trimethylsilylethynylphenyl)benzene



286b

1-Bromo-2-(trimethylsilylethynyl)benzene, **186** (3.28 g, 14.28 mmol) in THF (150 mL) was cooled to -78°C and *n*-BuLi (5.14 mL, 14.28 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (3.22 g, 14.28 mmol) in THF (75mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (400 mg, 0.34 mmol) and 1,3,5-tribromobenzene (1.00 g, 3.17 mmol) in THF (50 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction could be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a pale yellow solid (1.50 g, 83%) m.p. 156-157°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (s, 3H), 7.59 (dd, J = 6.5, 1.1 Hz, 3H), 7.52 (dd, J = 7.7, 1.0 Hz, 3H), 7.36 (dt, J = 6.2, 1.4Hz, 3H), 7.26 (dt, J = 7.6, 1.3 Hz, 3H), -0.04 (s, 27H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.8 (s), 139.2 (s), 133.6 (d), 129.5 (d), 129.3 (d), 128.6 (d), 126.8 (d), 121.4 (s), 104.8 (s), 97.7 (s), -0.49 (q); IR ($CHCl_3$): ν = 2959, 2151, 1581, 1481, 1419, 1250, 861 cm^{-1}; MS (FAB) *m/z* 595 (M^++H), 522, 404, 338, 312, 246, 219, 186, 137, 107, 73, 45.

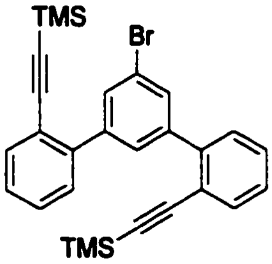
(2-Bromo-phenyl)-propynoic acid methyl ester



290

n-BuLi (4.8 mL, 11.9 mmol) was added to methylpropiolate, **293** (1.00 g, 11.9 mmol) in THF (20 mL) at -78°C and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnCl_2$ (23.8 mL, 0.5 M in THF) at -78°C was transferred via canula to the solution of the alkynyllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (300 mg, 0.259 mmol) and 1-iodo-2-bromobenzene (152 mL, 1.18 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction could be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a yellow oil (256 mg, 10%); yellow liquid; ^{1}H NMR (200 MHz, CDCl$_3$) δ 7.53 (m, *2*H), 7.24 (m, *2*H), 3.79 (s, *3*H); ^{13}C NMR (50 MHz, CDCl$_3$) δ 153.9 (s), 134.6 (d), 132.6 (d), 131.6 (d), 127.1 (d), 126.3 (s), 121.8 (s), 84.0 (s), 83.8 (s), 52.8 (s); IR (neat): *ν* = 2953, 2228, 1711, 1560, 1468, 1179 cm^{-1}; MS (EI) *m/z* 240, 238 (M$^+$), 208, 182, 161, 146, 118, 102, 76, 63, 51; HRMS calcd for $C_{10}H_7BrO_2$ 237.9629, found 237.9611.

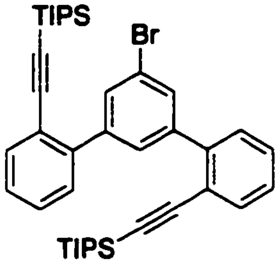
1-Bromo-3,5-(*o*-trimethylsilylethynylphenyl)benzene



295

1-Bromo-2-(trimethylsilylethynyl)benzene, **186** (813 uL, 3.80 mmol) in THF (20 mL) was cooled to -78°C and *n*-BuLi (1.54 mL, 3.80 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (857 mg, 3.80 mmol) in THF (20mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (250 mg, 0.216 mmol) and 1,3,5-tribromobenzene (600 mg, 1.90 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a colourless oil (1.45 g, 50%); ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.83 (d, *J* = 1.5 Hz, 1H), 7.74 (t, *J* = 1.5 Hz, 2H), 7.57 (dd, *J* = 7.7, 1.3 Hz, 2H), 7.39 (m, 4H), 7.28 (m, 2H), 0.11 (s, 18H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 142.3 (s), 141.6 (s), 133.6 (d), 131.1 (d), 129.3 (d), 129.0 (d), 128.7 (d), 127.4 (d), 121.4 (s), 121.0 (s), 104.1 (s), 98.3 (s), -0.30 (q); IR (CHCl$_3$): ν = 3062, 2959, 2897, 2157, 1570, 1484, 1249 cm^{-1}; MS (EI) *m/z* 500 (M$^+$), 421, 399, 333, 289, 235, 143, 73, 45; HRMS Calcd for $C_{28}H_{29}BrSi_2$ 500.0992, found 500.0975.

5'-Bromo-2,2"-bis-[(triisopropylsilanyl)-ethynyl]-[1,1';3',1"]terphenyl

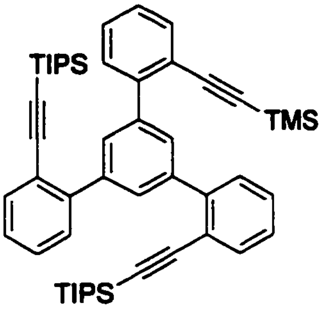


297

1-Bromo-2-(triisopropylsilylethynyl)benzene (3.00 g, 8.90 mmol) in THF (20 mL) was cooled to -78°C and *n*-BuLi (3.56 mL, 2.5 M) was added and the resulting yellow solution allowed to stir for 30 minutes. A solution of ZnBr$_2$ (2.00 g, 8.90 mmol) in THF (20 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of Pd(PPh$_3$)$_4$ (450 mg, 0.39 mmol) and 1,3,5-tribromobenzene (1.40 g, 4.44 mmol) in THF (20 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a yellow semi-solid. Alternatively, the reaction could be quenched by the addition of silica gel. Evaporating to dryness gives a crude yellow powder. Either crude product can be further purified by chromatography to give the product as a pale yellow solid (1.55 g, 70%) the product as a yellow gum (xx mg, xx%); ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.88 (t, *J* = 1.6 Hz, 1H), 7.72 (d, *J* = 1.5 Hz, 2H), 7.58 (m, 2H), 7.35 (m, 4H), 7.28 (m, 2H), 0.93 (s, 42H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 142.0 (s), 141.9 (s), 134.1 (d), 130.9 (d), 129.5 (d), 128.9 (d), 128.4

(d), 127.3 (d), 121.8 (s), 121.5 (s), 105.8 (s), 98.4 (s), 18.5 (q), 11.1 (d); IR ($CHCl_3$): ν = 3026, 2942, 2864, 2154, 1591, 1570, 1404, 1243, 995, 882 cm^{-1}; MS (FAB) *m/z* 627, 399, 289, 157; Anal Calcd for $C_{40}H_{53}Si_2Br$: C, 71.69; H, 7.97. Found: C, 71.76; H, 7.79.

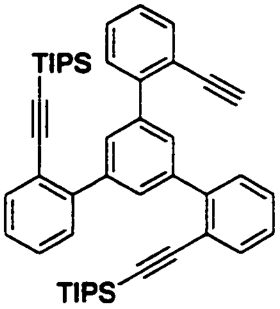
1,3-(*o*-Triispropylsilylethynylphenyl)-5-(*o*-trimethylsilylethynylphenyl)-benzene



298

1-Bromo-2-(trimethylsilylethynyl)benzene, **186** (520 mg, 2.04 mmol) in THF (20 mL) was cooled to -78°C and *n*-BuLi (820 μL, 2.04 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (460 mg, 2.04 mmol) in THF (10 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (300 mg, 0.26 mmol) and **297** (685 mg, 1.03 mmol) in THF (10 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction could be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a yellow viscous gum that produced a foam when subjected to vacuum (740 mg, 95%). ^1H NMR (500 MHz, $CDCl_3$) δ 8.05 (t, *J* = 1.6 Hz, 1H), 7.99 (d, *J* = 1.6 Hz, 2H), 7.70 (m, 1H), 7.57 (m, 2H), 7.45 (m, 3H), 7.34 (m, 1H), 7.26 (m, 1H), 0.82 (s, 42H), -0.10 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.3 (s), 139.6 (s), 134.1 (d), 133.9 (d), 129.8 (d), 129.7 (d), 129.7 (d), 129.1 (d), 128.8 (d), 128.4 (d), 126.9 (d), 126.8 (d), 128.5 (d), 126.9 (d), 126.8 (d), 121.8 (s), 120.6 (s), 106.5 (s), 94.3 (s), 83.3 (s), 80.2 (d), 18.4 (q), 11.1 (d), -0.49 (q); IR (neat): ν = 3060, 2891, 2155, 1590, 1460, 1208, 843 cm^{-1}; MS (ES) *m/z* 780 (M+NH$^+$), 587, 551, 495, 454, 413, 393, 364, 349, 342.

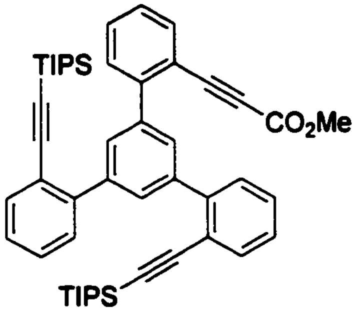
1,3-(*o*-Triispropylsilylethynylphenyl)-5-(*o*-ethynylphenyl)-benzene



299

Compound **298** (333 mg, 0.43 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a yellow viscous gum (920 mg, 90%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.12 (t, *J* = 1.6 Hz, 1H), 7.90 (d, *J* = 1.6 Hz, 2H), 7.64 (m, 2H), 7.49 (m, 2H), 7.34 (m, 4H), 3.03 (s, 1H), 0.86 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 144.3 (s), 143.6 (s), 139.6 (s), 134.1 (d), 133.9 (d), 129.8 (d), 129.7 (d), 129.7 (d), 129.1 (d), 128.8 (d), 128.4 (d), 126.9 (d), 126.8 (d), 128.5 (d), 126.9 (d), 126.8 (d), 121.8 (s), 120.6 (s), 106.5 (s), 94.3 (s), 83.3 (s), 80.2 (d), 18.4 (q), 11.1 (d); IR (neat): *v* = 3060, 3026, 2859, 2153, 1590, 1465, 1365, 1160 cm^{-1}; MS (ES) *m/z* 691 (M+H$^+$), 587, 557, 481, 393, 388, 364, 349, 342.

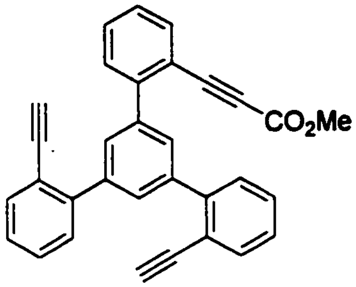
1-(*o*-Methylcarboxy-ethynylphenyl)-3,5-(*o*-triisopropylsilylethynylphenyl)-benzene



300

n-BuLi (550 uL, 1.22 mmol) was added to a solution of **299** (842 mg, 1.22 mmol) in THF (25 mL) at -78°C and the resulting yellow solution stirred for 15 minutes. To this solution was added methylchloroformate (0.10 mL, 1.28 mmol) the solution was allowed to come to RT and stir overnight. The solution was poured into ether and washed with water and brine, dried and evaporated to yield a slightly yellow oil. The oil was purified by purified by chromatography to give the product as a clear gum (818 mg, 90%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.10 (t, *J* = 1.6 Hz, 1H), 7.90 (d, *J* = 1.6 Hz, 2H), 7.60 – 7.69 (m, 6H), 7.30 – 7.57 (m, 6H), 3.62 (s, 3H), 0.87 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 154.1 (s), 143.4 (s), 140.0 (s), 138.5 (s), 134.7 (s), 134.0 (d), 130.8 (d), 130.0 (d), 129.8 (d), 129.0 (d), 128.3 (d), 127.0 (d), 126.8 (d), 121.7 (s), 117.9 (s), 106.4 (s, two overlapping signals), 94.2 (s), 86.5 (s), 83.0 (s), 52.5 (q), 18.3 (q), 11.0 (d); IR (neat): ν = 2942, 2863, 2153, 1718, 1655, 1412, 1295, 1199 cm^{-1}; MS (ES) *m/z* 766 ($M+NH_4^+$), 561, 345, 197, 159, 145, 73, 71.

1-(*o*-Methylcarboxy-ethynylphenyl)-3,5-(*o*-ethynylphenyl)-benzene

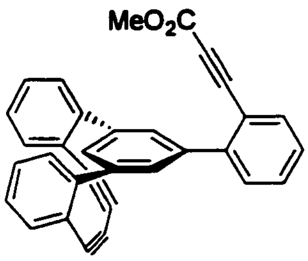


301

Compound **300** (130.2 mg, 0.22 mmol) was dissolved in THF (20 mL) and MeOH (1 mL) and TBAF (1.03 mL, 1.0 M) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was further purified by chromatography to give the product as a yellow oil (450 mg, 99%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.19 (t, *J* = 1.5 Hz, 1H), 7.85 (d, *J* = 1.5 Hz, 2H), 7.74 – 7.31 (m, 12H), 3.71 (s, 3H), 3.12 (s, 2H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 154.2 (s), 145.1 (s), 143.9 (s), 139.8 (s), 138.6 (s), 134.7 (d), 133.8 (d), 130.8 (d), 129.8 (d, 2 overlapping signals), 129.7 (d), 129.0 (d), 128.9 (d), 127.2 (d), 127.1 (d), 120.4 (s), 117.8

(d), 86.5 (s), 83.2 (s), 83.0 (s), 80.5 (d), 52.5 (q); IR (neat): ν = 3290, 3058, 2942, 2221, 1719, 1590, 1274 cm^{-1}; MS (EI) *m/z* 436 (M$^+$), 403, 376, 350, 274, 187, 29; HRMS calcd for $C_{32}H_{20}O_2$, 436.1464, found 436.1444.

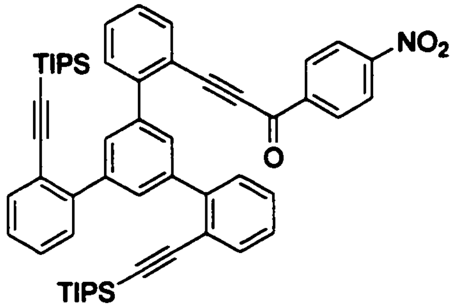
1,3-Diyne-[4](2,2")[1,1';3',1"]-(5'-*o*-(methylcarboxyethynyl)phenyl)terphenylophane



302

Cu(OAc)$_2$ (1.64 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (160 mL) and **301** (444 mg, 1.01 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (2:1 Pet Ether: Et_2O) to yield the product as a yellow semi-solid (372 mg, 85%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.39 (t, *J* = 1.7 Hz, 1H), 7.75 (m, 4H), 7.53 – 7.22 (m, 10H), 3.80 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 154.2 (s), 148.4 (s), 144.8 (s), 139.7 (d), 138.3 (s), 137.2 (d), 134.8 (d), 130.9 (d), 130.8 (d), 130.4 (d), 129.8 (d), 129.7 (d), 127.5 (d), 127.2 (d), 121.6 (s), 117.7 (s), 107.8 (s), 87.0 (two overlapping signals, s), 86.4 (s), 82.9 (s), 52.6 (q); IR (neat): ν = 3290, 2942, 2221, 1759, 1590, 740 cm^{-1}; MS (ES) *m/z* 435 (M$^+$), 402, 375, 327, 180, 148, 72, 55.

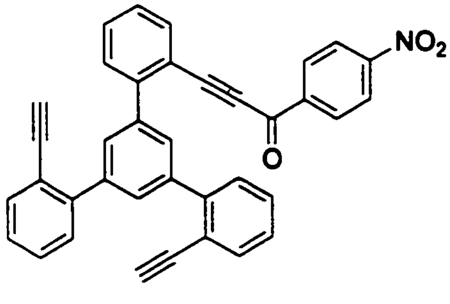
1-(*o*-(*p*-nitrophenylethynylketone)phenyl)-3,5-(*o*-triisopropylsilylethynylphenyl)-benzene



303

n-BuLi (384 uL, 0.96 mmol) was added to a solution of **299** (663 mg, 0.96 mmol) in THF (20 mL) at -78°C and the resulting yellow solution stirred for 15 minutes. To this solution was added 4-nitrobenzoyl chloride (187 mg, 1.01 mmol) the solution was allowed to come to RT and stir overnight. The solution was poured into ether and washed with water and brine, dried and evaporated to yield a slightly yellow oil. The oil was purified by chromatography (Pet. Ether) to provide the product as a yellow liquid (500 mg, 62%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.06 (m, 2H), 7.96 - 7.82 (m, 5H), 7.64 – 7.53 (m, 5H), 7.45 – 7.24 (m, 7H), 1.20 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 175.7 (s), 150.4 (s), 145.2 (s), 142.5 (s), 140.6 (s), 140.3 (s), 139.1 (s), 135.6 (d), 134.5 (d), 131.4 (d), 130.4 (d), 130.2 (d), 129.9 (d), 129.5 (d), 128.9 (d), 128.7 (d), 127.4 (d), 127.3 (d), 123.6 (d), 121.7 (s), 118.0 (s), 106.1 (s), 95.2 (s), 94.7 (s), 89.3 (s); IR (neat): ν = 3063, 2943, 2863, 2154, 1727, 1646, 1530, 1411, 1202, 760 cm^{-1}; MS (ES) *m/z* 857 ($M+NH_4^+$), 554, 447, 421, 340, 323, 250, 234, 135, 121, 93, 72, 54.

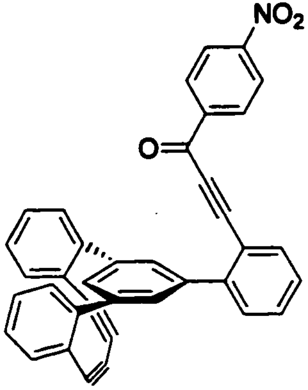
1-(*o*-(*p*-nitrophenylethynylketone)phenyl)-3,5-(*o*-ethynylphenyl)-benzene



304

Compound **303** (982 mg, 1.16 mmol) was dissolved in THF (15 mL) and TBAF (0.575 mL, 1.0 M) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was further purified by chromatography to give the product as a yellow oil (612 mg, 99%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.21 – 7.82 (m, 6H), 7.67 – 7.27 (m, 13H), 3.18 (s, 2H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 163.9 (s), 165.0 (s), 147.8 (s), 147.2 (s), 145.5 (d), 143.6 (s), 140.2 (s), 139.5 (d), 134.1 (d), 132.0 (d), 130.9 (d), 130.3 (d), 129.8 (d), 129.7 (d), 129.5 (d), 129.2 (d), 127.4 (d), 127.2 (d), 123.7 (d), 120.3 (s), 119.5 (s), 90.1 (s), 88.5 (s), 83.5 (s), 80.4 (d); IR (neat): v = 3288, 3062, 2863, 2193, 1646, 1593, 1522, 1346, 1109, 760 cm^{-1}; MS (FAB) *m/z* 510, 463, 363, 186, 136, 59.

1,3-Diyne-[4](2,2")[1,1';3',1"]-(5'-*o*-(*p*-nitrophenylethynylketone)phenyl)terphenylophane

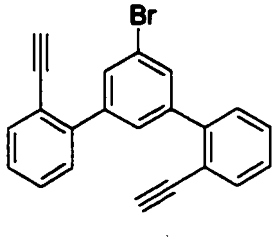


305

$Cu(OAc)_2$ (460 mg, 6 equiv.) was dissolved in pyridine: ether (3:1) (180 mL) and **304** (150 mg, 0.28 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed excessively with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (2:1 Pet Ether: Et_2O) to yield the product as a yellow solid (80 mg, 53%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (br, 2H), 7.87 (s, 3H), 7.67 (m, 3H), 7.45 (m, 5H), 7.34 (m, 2H), 7.20 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.7 (s), 147.9 (s), 147.1 (s), 145.4 (s), 144.3 (s), 139.7 (s), 139.6 (s), 134.2 (d), 131.9 (d), 131.1

(d), 130.5 (d), 129.8 (d), 129.7 (d), 129.6 (d), 129.6 (d), 127.5 (d), 127.3 (d), 127.2 (d), 123.7 (d), 119.9 (s), 119.4 (s), 90.0 (s), 88.5 (s), 82.6 (s), 77.2 (s); IR (neat): $v = 3064$, 2923, 2852, 2221, 1696, 1635, 1419, 1338, 1042 cm^{-1}; MS (ES) *m/z* 1070 ($2M+NH_4^+$), 361, 323, 252, 240, 144, 135, 107, 72.

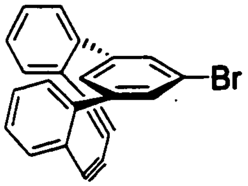
1-Bromo-3,5-(*o*-ethynylphenyl)benzene



306

Compound **295** (310 mg, 0.61 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a yellow gum (220 mg, 99%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.89 (t, J = 1.5 Hz, 2H), 7.74 (d, J = 1.5 Hz, 4H), 7.65 (m, 2H), 7.40 (m, 6H), 3.13 (s, 2H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 142.6 (s), 141.6 (s), 133.9 (d), 131.1 (d), 129.4 (d), 129.1 (d), 128.9 (d), 127.5 (d), 121.4 (s), 121.4 (s), 82.7 (s), 80.9 (d); IR ($CHCl_3$): $v = 3289$, 3062, 2957, 2105, 1592, 1405, 1249, 865 cm^{-1}; MS (EI) *m/z* 356 (M^+), 330, 303, 289, 276, 250, 224, 200, 74, 138, 125, 73; HRMS Calcd for $C_{22}H_{13}Br$ 256.0261, found 356.0036.

1,3-Diyne-[4](2,2")[1,1';3',1"]-5'-bromoterphenylophane



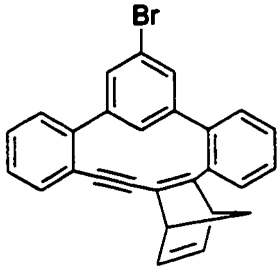
307

$Cu(OAc)_2$ (1.00 g, 6 equiv.) was dissolved in pyridine: ether (3:1) (400 mL) and **306** (220 mg, 0.61 mmol) in 20 mL of pyridine:ether (3:1) was added over 3 hr *via* syringe pump.

The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (Pet Ether) to yield the product as an off white solid (263 mg, 60%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.25 (m, 1H), 7.52 (m,2H), 7.42 (d, *J*= 2.5 Hz, 2H), 7.30 (m,6H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 147.2 (s), 141.7 (s), 136.5 (d), 132.6 (d), 130.0 (d), 129.9 (d), 127.9 (d), 127.5 (d), 121.8 (s), 121.4 (s), 107.4 (s), 87.1 (s); IR ($CHCl_3$): *ν* = 3048, 2918, 2875, 2201, 1604, 1486, 1455, 897, 753, 679 cm^{-1}; MS (FAB) *m/z* 354 (M^+), 330, 274, 248, 137, 73; HRMS Calcd for $C_{22}H_{11}Br$ 354.0044, found 353.9810.

Crystal size 0.40 x 0.10 x 0.10 mm^3, triclinic, space group *P*-1, scan range $3.8<2\theta<57.86°$, a=3.9174(4), b=10.510(1), c=18.179(2) Å, ß=92,290(2)°, *V* =745.7(1) Å^3, Z=2, ρ_{calcd}=1.582 g cm-3, u=2.752 mm^{-1}, 3433 unique reflections at -80°C, of which 5844 were taken as observed [$I_o>2.00\sigma(I)$], R=0.0531, R_w=0.0993. Crystallographic data (excluding structure factors) for this structure has been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-162163. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

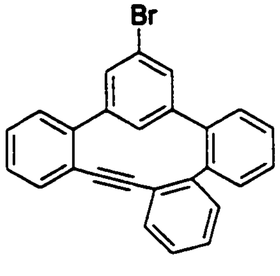
1,3-Diyne-[4](2,2")[1,1';3',1"]-5'-bromoterphenylophane Cyclopentadiene Diels-Alder Adduct



310

A sealed tube was charged with **307** (100 mg, 0.28 mmol) and freshly distilled cyclopentadiene (150 mg, 2.25 mmol) and dissolved in toluene (10 mL). The solution was heated to 120 °C and allowed to stir in a closed system for 15 hr. The solution was cooled to room temperature and transferred to a round bottom flask. Silica gel was added and the solvent removed under reduced pressure. The resulting crude solid was purified by chromatography (Pet. Ether) to give the product as a bright yellow solid (32 mg, 28%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.9 Hz, 1H), 8.41 (m, 1H), 7.78 (m, 2H), 7.52 (m, 3H), 7.01- 7.49 (m, 8H), 4.08 (s, 1H), 3.82 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) □ 152.4 (s), 148.6 (s), 141.8 (d), 141.2 (s), 140.1 (d), 137.7 (s), 135.9 (s), 135.6 (s), 134.0 (d), 132.3 (s), 132.3 (d), 131.9 (s), 131.8 (s), 128.8(d), 128.1 (s), 128.0 (d), 127.5 (d), 126.7 (d), 124.6 (d), 124.1 (d), 123.7 (d), 122.9 (d), 120.8 (s), 68.2 (t), 57.2 (d), 53.9 (d); IR (film): *v* = 2918, 2875, 2201, 1604, 897, 755, cm^{-1}; MS (FAB) *m/z* 420 (M$^+$), 393, 339, 313, 274, 211, 149, 97, 57; HRMS Calcd for $C_{27}H_{17}Br$ 420.0514, found 420.0516.

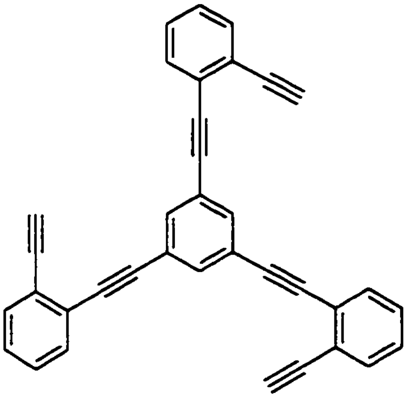
1,3-Diyne-[4](2,2")[1,1';3',1"]-5'-bromoterphenylophane 1,3-Cyclohexadiene Diels-Alder Adduct



311

A sealed tube was charged with **307** (100 mg, 0.28 mmol) and 1,3-cyclohexadiene (00 mg, 1.12 mmol) and dissolved in toluene (15 mL). The solution was heated to 120 °C and allowed to stir in a closed system for 15 hr. The solution was cooled to room temperature and transferred to a round bottom flask. Silica gel was added and the solvent removed under reduced pressure. The resulting crude solid was purified by chromatography (Pet. Ether) to give the product as a yellow solid (26 mg, 23%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 - 7.35 (m, 5H), 7.33 – 7.22 (m, 5H), 7.21 – 7.14 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9 (s), 142.8 (s), 141.9 (s), 139.8 (s), 138.2 (d), 134.3 (d), 134.1 (d), 133.6 (s), 133.5 (s), 133.0 (d), 129.7 (d), 128.7 (d), 128.4 (d), 128.3 (d), 128.0 (s), 127.9 (d), 127.6 (d), 127.4 (d), 126.8 (d), 125.6 (d), 123.8 (d), 122.7 (d), 121.7 (s), 120.4 (s), 97.6 (s), 94.5 (s); IR (film): *v* = 2918, 2875, 2201, 1604, 897, 755, cm^{-1}; MS (EI) *m/z* 406 (M$^+$) 326, 270, 250, 203, 163, 143, 97, 57; HRMS Calcd for $C_{26}H_{15}Br$ 406.0357, found 406.0363.

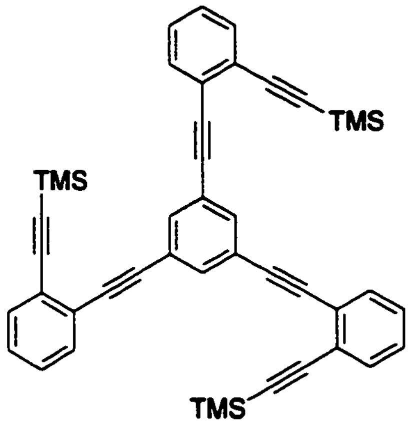
1,3,5-((*o*-Ethynylphenyl)ethynyl)benzene



313

Compound **316** (880 mg, 1.32 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography gave the product as a white solid (500 mg, 84%). m.p. 106-108°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (s, 3H), 7.55 (m, 6H), 7.33 (dt, J = 7.6, 1.6 Hz, 3H), 7.29 (dt, J = 7.5, 1.5 Hz, 3H), 3.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.5 (d), 132.6 (d), 131.9 (d), 128.6 (d), 128.3 (d), 125.7 (s), 124.8 (s), 123.9 (s), 91.7 (s), 89.0 (s), 82.0 (s), 81.4 (d); IR ($CHCl_3$): ν = 3289, 3053, 2207, 2107, 1593, 1580, 1478, 1442, 1243, 1099, 876 cm^{-1}; MS (EI) *m/z* 450 (M$^+$), 420, 281, 224, 181, 149, 131, 91, 69, 44; HRMS Calcd for $C_{36}H_{18}$ 450.1409, found 450.1354.

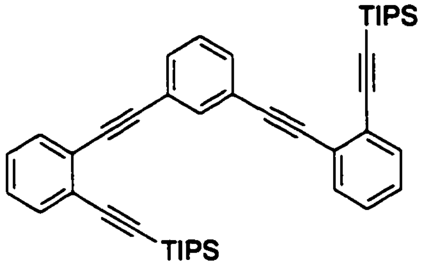
1,3,5-((*o*-Trimethylsilylethynylphenyl)ethynyl)benzene



316

CuI (10 mg, 0.05 mmol), $Pd(PPh_3)_4$ (19 mg, 0.03 mmol) and **317** (50 mg, 0.33 mmol) were added to a flame dried round bottom flask and dissolved in DEA (10 mL) and THF (10 mL). The resulting dark green solution was degassed for 15 minutes by sparging with Ar or N_2. **186** (333 mg, 1.00 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (5% Ethyl Acetate: Pet. Ether) to provide a slightly pale yellow solid (88 mg, 40%); m.p. 142-144°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (s, 3H), 7.50 (m, 6H), 7.29 (m, 6H), 0.29 (s, 27H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.3 (d), 132.3 (d), 131.6 (d), 128.2 (d), 128.2 (d), 126.0 (s), 125.6 (s), 124.1 (s), 103.3 (s), 99.0 (s), 91.7 (s), 89.4 (s), 0.07 (q); IR ($CHCl_3$): ν = 2958, 2902, 2158, 1580, 1476, 1444, 1249, 863, 842, 757 cm^{-1}; MS (FAB) *m/z* 667 (M^++H), 580, 491, 391, 289, 246, 219, 185, 133, 93.

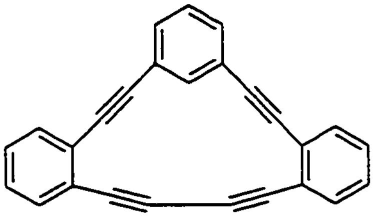
3,5-(*o*-Ethynylphenylethynyl)-benzene



320

Compound **319** (1.50 g, 5.32 mmol) in THF (100 mL) was cooled to -78°C and *n*-BuLi (2.33 mL, 5.32 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (1.20 g, 5.32 mmol) in THF (25mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (280 mg, 0.242 mmol) and 1,3-diiodobenzene (800 mg, 2.42 mmol) in THF (25 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are purified by silica gel chromatography (Pet. Ether) to yield the product as a bright yellow oil (940 mg, 61%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.54 (m, 6H), 7.31 (m, 5H), 1.81 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 135.2 (s), 132.8 (d), 132.1 (d), 131.6 (s), 131.4 (d), 128.4 (d), 128.1 (d), 128.0 (two overlapping signals, s), 123.6 (s), 105.3 (s), 95.2 (s), 92.3 (s), 88.8 (s), 18.7 (q), 11.4 (d); IR (neat) ν = 3060, 2864, 2155, 1815, 1595, 1365, 1209, 1071 cm^{-1}; MS (FAB) *m/z* 639 (M^++H), 595, 553, 515, 459, 415, 373, 345, 302, 279, 243, 180, 157, 115.

3,9-Di-*o*-phenyl-1,5,7,11-tetrayne-[12]metacyclophane

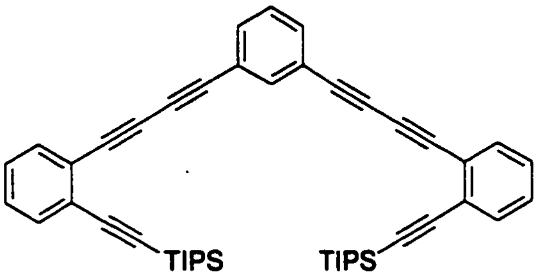


322

Compound **320** (940 mg, 1.47 mmol) was dissolved in THF (20 mL) and TBAF (3.10 mL, 1.0 M) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was quickly passed through a column of silica gel flash (Pet Ether) and gave the product as a yellow oil (430 mg, 90%). The desilylated product (162 mg, 0.49 mmol) was added to a solution of

$CuOAc_2$ (540 mg, 6 equiv.) in pyridine: ether (3:1) (280 mL) in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solid can be further purified by chromatography (9:1 Pet Ether: Et_2O) to yield the product as an orange solid (96 mg, 60%); ^1H NMR (500 MHz, $CDCl_3$) δ 8.69 (s, 1H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.41 (d, *J* = 7.7Hz, 2H), 7.29 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0 (d), 132.7 (d), 129.8 (d), 129.1 (d), 128.8 (d), 128.1 (d), 127.4 (d), 127.1 (s), 126.3 (s), 124.0 (s), 95.8 (s), 92.2 (s), 82.3 (s), 78.4 (s); IR (film) ν = 3219, 2346, 1648, 1550, 1400, 1115, 931 cm^{-1}; MS (EI) *m/z* 324 (M^+), 296, 270, 161, 97, 71, 57; HRMS Calcd for $C_{26}H_{12}$, 324.0939, found 324.0919.

1,3-((*o*-Triisopropylsilylethynylphenyl)butadiynyl)benzene

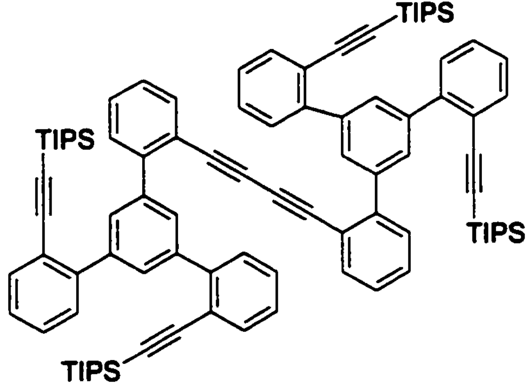


323

CuI (2 mg, 0.01 mmol), $Pd_2(dba)_3$ (10 mg, 0.01 mmol) and 1,3-(bromoethynyl)benzene (120 mg, 0.43 mmol) were added to a flame dried round bottom flask and dissolved in benzene (20 mL). The solution was degassed for 15 minutes by sparging with Ar or N_2. **319** (333 mg, 1.00 mmol) followed by 1,2,2,6,6-pentamethylpiperidine (133 mg, 0.86 mmol) were added and the solution stirred at ambient temperature overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (5:1 Pet. Ether: Et_2O) to provide a yellow oil that partially solidifies on standing (114 mg, 40%); ^1H NMR (500 MHz, $CDCl_3$) δ 7.60 (t, *J*= 1.2 Hz, 1H), 7.50 (m, 6H), 7.28 (m, 5H), 1.18 (s, 42H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.7 (d), 132 (d), 132.6 (d), 132.3 (d), 128.8 (d), 128.7 (d), 127.9 (d), 127.4 (s), 124.6

(s), 122.6 (s), 104.5 (s), 96.2 (s), 80.9 (s), 80.8 (s), 77.4 (s), 74.8 (s), 18.7 (q), 11.3 (d); IR (neat) ν =2942, 2219, 2157, 1589, 1468, 1283, 1245, 1072 cm^{-1}; MS (CI) *m/z* 686 (M^+) 643, 601, 475, 427, 300, 251, 195, 157, 115; Anal Calcd for $C_{48}H_{54}Si_2$: C, 83.90; H, 7.92. Found: C, 84.00; H, 7.82.

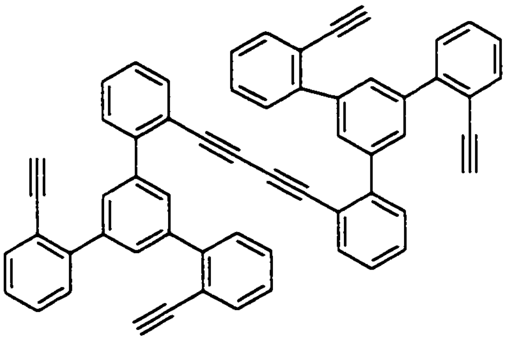
1,4-Di-(*o*-(3,5-(*o*-triispropylsilylethynylphenyl)phenylbutadiyne



325

$Cu(OAc)_2$ (196 mg, 3 equiv.) was dissolved in pyridine: ether (3:1) (220 mL) and **299** (250 mg, 0.36 mmol) was added in 20 mL of pyridine:ether (3:1) over 3 hr *via* syringe pump. The blue solution gradually became emerald green. Once addition was complete, the cooled solution was poured into ether and 1 M HCl. The organic phase was washed with 1 M HCl until all pyridine was removed, and brine, dried and evaporated to yield a yellow solid. Alternatively, silica gel can be added to the reaction mixture and the solvent removed under reduced pressure. The crude solids can be further purified by chromatography (20:1 Pet Ether: Et_2O) to yield the product as a yellow gum that foams under vacuum (263 mg, 60%); ^1H NMR (200 MHz, $CDCl_3$) δ 8.09 (t, J = 1.4 Hz, 1H), 7.87 (d, J = 1.4 Hz, 2H), 7.58 (m, 7H), 7.28 (m, 5H), 0.86 (s, 84H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 144.7 (s), 143.5 (d), 139.8 (d), 139.2 (s), 134.3 (s), 134.0 (d), 133.0 (d, 2 overlapping signals), 129.7 (s), 129.1 (s), 128.9 (d), 128.5 (d), 126.9 (d), 126.8 (d), 121.7 (d), 120.4 (s), 106.6 (s), 94.1 (s), 82.0 (s), 77.3 (s), 18.4 (q), 11.1 (d); IR (neat): ν = 2942, 2864, 2154, 1590, 1485, 882 cm^{-1}; MS (ES) *m/z* 1397 ($M+NH_4^+$), 1135, 1119, 903, 887, 882, 865, 855, 797, 775, 751.

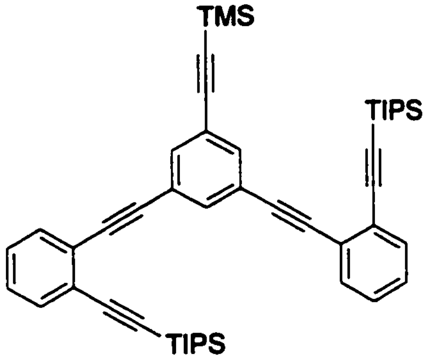
1,4-Di-(*o*-(3,5-(*o*-ethynylphenyl)phenylbutadiyne



326

Compound **325** (1.86 g, 1.34 mmol) was dissolved in THF (250 mL) and TBAF (5.46 mL, 1.0 M) was added and the resulting solution allowed to stir at RT until no starting material remained by TLC. The reaction mixture was separated between ether and water and the ether phase washed with water and brine and dried. Silica gel was added and the mixture evaporated to dryness to yield a crude solid. The solid was further purified by chromatography (Pet Ether) to yield the product as a yellow gum (950 mg, 61%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.91 (s, 4H), 7.85 (s, 2H), 7.58 (m, 14H), 7.30 (m, 10H), 3.09 (s, 6H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 144.6 (s), 144.0 (d), 139.8 (d), 139.2 (s), 134.3 (s), 133.7 (d), 129.9 (d), 129.8 (s), 129.5 (d), 129.4 (s), 129.1 (d), 129.0 (d), 127.2 (d), 127.0 (d), 127.4 (d), 120.2 (s), 83.2 (s), 82.3 (s), 80.6 (d), 77.4 (s); IR (neat): ν = 2930, 2874, 1651, 1485, 1380, 1134, 892, 761 cm^{-1}; MS (ES) *m/z* 772 ($M+NH_4^+$), 278, 242, 185, 141, 129, 72, 54.

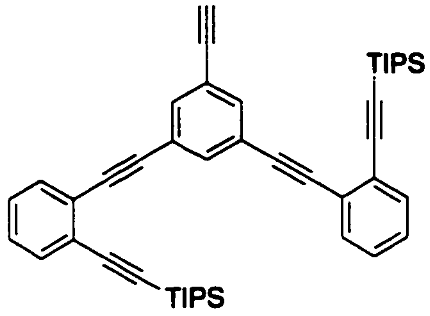
1,3-((*o*-Trimethylsilylethynylphenyl)ethynyl)-5-(trimethylsilylethynyl)benzene



339

CuI (100 mg, 0.52 mmol), $Pd(PPh_3)_2Cl_2$ (100 mg, 0.15 mmol) and **338** (1.10 g, 3.35 mmol) were added to a flame dried round bottom flask and dissolved in DEA (50 mL) and THF (50 mL). The resulting dark green solution was degassed for 15 minutes by bubbling through a stream of Ar or N_2. Compound **319** (1.90 g, 6.73 mmol) was added and the solution brought to reflux overnight. Silica gel was added and the solvent was then removed under reduced pressure and the resulting crude solid was purified by chromatography (Pet. Ether) to provide the product as a yellow gum (600 mg, 25%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46- 7.60 (m, 8H), 7.32 (m, 3H), 1.12 (s, 42H), 0.23 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.1 (d), 133.7 (d), 132.8 (d), 131.9 (d), 128.3 (d), 128.0 (s), 127.7 (s), 126.1 (s), 125.1 (s), 121.6 (d), 105.0 (s), 102.4 (s), 96.4 (s), 95.5 (s), 90.6 (s), 90.0 (s), 18.62 (q), 11.31 (d), -0.27 (q); IR (neat): ν = 3062, 2944, 2864, 2723, 2217, 2155, 1584, 1365, 1250, 1036 cm^{-1}; MS (ES) m/z 720, 635, 533, 497, 453, 409, 387, 386, 349, 327, 305, 278, 232.

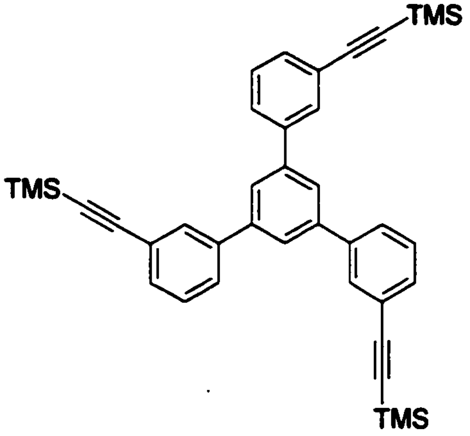
1,3-((*o*-Trimethylsilylethynylphenyl)ethynyl)-5-ethynylbenzene



340

Compound **339** (540 mg, 0.73 mmol) was dissolved in a minimum amount of THF (10 mL) and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a clear oil (360 mg, 75%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.69 (t, J = 1.6 Hz, 1H), 7.56 (m, 5H), 7.28 (m, 4H), 3.16 (s, 1H), 1.19 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 134.6 (d), 134.4 (d), 133.7 (d), 132.8 (d), 132.0 (d), 128.3 (d), 128.0 (d), 126.1 (s), 125.3 (s), 124.9 (s), 124.0 (s), 121.7 (s), 105.0 (s), 95.4 (s), 90.5 (s), 90.3 (s), 81.2 (s), 79.0 (d), 77.0 (s), 18.6 (q), 11.3 (d); IR (neat): ν = 3300, 2942, 2155, 1586, 1554, 1214, 1159, 1036, 882 cm^{-1}; MS (ES) *m/z* 402, 384, 366, 349, 277; Anal Calcd for $C_{46}H_{54}Si_2$: C, 83.32; H, 8.21. Found: C, 83.53; H, 8.22.

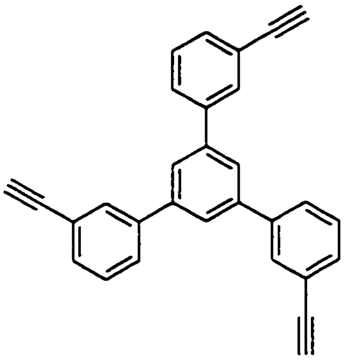
1,3,5-(*m*-Trimethylsilylethynylphenyl)benzene



342

1-Bromo-3-(trimethylsilylethynyl)benzene, **236** (2.89 g, 11.4 mmol) in THF (150 mL) was cooled to -78°C and *n*-BuLi (5.6 mL, 11.4 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (2.56 g, 11.4 mmol) in THF (50mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (350 mg, 0.303 mmol) and 1,3,5-tribromobenzene (800 mg, 2.53 mmol) in THF (75 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a white solid (1.1 g, 74%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.75 (m, 6H), 7.25 (m, 3H), 7.52 (m, 6H), 0.26 (s, 27H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 141.6 (s), 140.9 (s), 131.1 (d), 130.8 (d), 128.8 (d), 127.5 (d), 125.2 (d), 123.8 (s), 104.9 (s), 94.6 (s), -0.04 (q); IR ($CHCl_3$): ν = 2936, 2855, 2159, 1585, 1560, 1253, 1126 cm^{-1}; MS (FAB) *m/z* 595 (M^++H), 538, 491, 375, 335, 279, 226, 136.

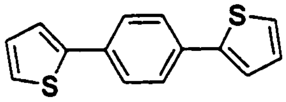
1,3,5-(*m*-Ethynylphenyl)benzene



343

Compound **342** (1.1 g, 1.85 mmol) was dissolved in a minimum amount of THF and MeOH (1/4 the amount of THF) and H_2O (drops) and a large excess of K_2CO_3 was added. The solution was stirred vigorously for until no starting material remained by TLC. The mixture was then separated between pentane and water. The organic phase was washed with water, dried and evaporated to yield a crude product. Purification by chromatography yielded the product as a yellow oil (587 mg, 84%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.82 (m, 3H), 7.74 (m, 3H), 7.65 (m, 3H), 7.47 (m, 6H), 3.12 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 141.5 (s), 141.0 (s), 131.3 (d), 131.0 (d), 128.9 (d), 127.8 (d), 125.3 (d), 122.8 (s), 83.5 (s), 77.6 (d); IR ($CHCl_3$): ν = 2925, 2861, 2343, 1655, 1560, 1113 cm^{-1}; MS (EI) *m/z* 378 (M^+), 332, 276, 220, 189, 162, 143, 112, 84, 44; HRMS calcd for $C_{30}H_{18}$ 378.1409, found 378.1432.

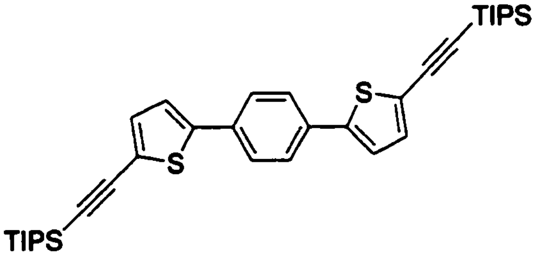
1,4-Di-(2'-thiophenyl)benzene



364

2-Bromothiophene, **359** (600 uL, 6.13 mmol) in THF (30 mL) was cooled to -78°C and *n*-BuLi (3.04 mL, 6.13 mmol) was added and the resulting yellow solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (1.38 g, 6.13 mmol) in THF (20mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (300 mg, 0.259 mmol) and 1,4-dibromobenzene (482 mg, 2.04 mmol) in THF (20 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a tan solid (400 mg, 82%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (s, 4H), 7.32 (dd, J = 3.6, 1.1 Hz, 2H), 7.27 (dd, J = 5.1, 1.1 Hz, 2H), 7.07 (dd, J = 5.1, 3.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.9 (s), 133.5 (s), 128.1 (d), 126.3 (d), 124.8 (d), 123.1 (d); IR ($CHCl_3$): ν = 2859, 1570, 1484, 740 cm^{-1}; MS (EI) m/z 242 (M^+), 197, 162, 143, 121, 95, 57; HRMS Calcd for $C_{14}H_{10}S_2$ 242.0237, found 242.0034.

1,4-Di-((5'-triisopropylsilylethynyl)-2'-thiophenyl)benzene



371

Compound **368** (325 mg, 1.23 mmol) in THF (10 mL) was cooled to -78°C and n-BuLi (610 uL, 1.23 mmol) was added and the resulting green solution allowed to stir for 15 minutes. A solution of $ZnBr_2$ (277 mg, 1.23 mmol) in THF (5 mL) at -78°C was transferred via canula to the solution of the aryllithium and the solution warmed to 0°C and allowed to stir for 15 minutes. A solution of $Pd(PPh_3)_4$ (100 mg, 0.086 mmol) and 1,4-dibromobenzene (100 mg, 0.42 mmol) in THF (5 mL) was then transferred via canula to the solution and warmed to RT and then refluxed overnight (~15 hr). The reaction mixture was then quenched with saturated aqueous ammonium chloride and separated between ether and water. The ether phase was washed with water and brine. The organic layer was dried and evaporated to yield a dark yellow semi-solid. Alternatively the reaction can be quenched by addition of silica gel. The crude mixture is then evaporated to dryness on a rotary evaporator. The crude products are further purified by chromatography to give the product as a bright yellow solid (210 mg, 92%); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.73 (s, 4H), 7.61 (m, 1H), 7.49 (m, 1H), 1.10 (s, 42H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 143.9 (s), 141.6 (s), 133.5 (s), 128.1 (d), 126.3 (d), 124.8 (d), 104.9 (s), 94.6 (s), 18.7 (q), 11.3 (d); IR ($CHCl_3$): ν = 2865, 2140, 1560, 1460, 1248, 919 cm^{-1}; MS (EI) *m/z* 602 (M^+), 559, 526, 483, 428, 385, 309, 248, 152, 121, 77, 39; HRMS Calcd for $C_{36}H_{50}S_2Si_2$ 603.2895, found 603.2946.

CHAPTER 8: APPENDIX X-RAY CRYSTALLOGRAPHIC DATA.

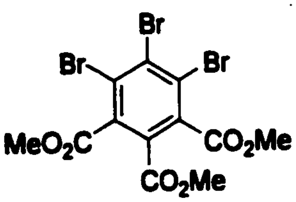


Table 1. Crystal data and structure refinement for 1.

Identification code	af001
Empirical formula	C12 H9 Cl3 O6
Formula weight	355.54
Temperature	294(2) K
Wavelength	0.71073 A
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	a = 13.6271(7) A alpha = 90 deg. b = 5.8195(3) A beta = 109.090(1) de c = 19.6635(9) A gamma = 90 deg.
Volume, Z	1473.62(13) A^3, 4
Density (calculated)	1.603 Mg/m^3
Absorption coefficient	0.644 mm^-1
F(000)	720
Crystal size	0.40 x 0.20 x 0.20 mm
Theta range for data collection	1.58 to 28.69 deg.
Limiting indices	-13<=h<=18, -7<=k<=7, -24<=l<=25
Reflections collected	8767
Independent reflections	3446 [R(int) = 0.0265]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3446 / 0 / 190
Goodness-of-fit on F^2	1.023
Final R indices [I>2sigma(I)]	R1 = 0.0369, wR2 = 0.1027
R indices (all data)	R1 = 0.0424, wR2 = 0.1104
Largest diff. peak and hole	0.332 and -0.286 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	2177(1)	1571(1)	1746(1)	49(1)
Cl(2)	772(1)	4957(1)	2222(1)	55(1)
Cl(3)	620(1)	4660(1)	3756(1)	54(1)
O(1)	1461(2)	-842(3)	4798(1)	65(1)
O(2)	2211(1)	2539(3)	5199(1)	62(1)
O(3)	3667(1)	-1296(3)	4967(1)	70(1)
O(4)	3491(1)	-3870(3)	4093(1)	61(1)
O(5)	4459(1)	-960(3)	3262(1)	52(1)
O(6)	3284(1)	-3060(3)	2439(1)	75(1)
C(1)	2136(1)	1457(3)	2611(1)	35(1)
C(2)	1495(1)	2976(3)	2818(1)	35(1)
C(3)	1452(1)	2853(3)	3516(1)	35(1)
C(4)	2034(1)	1264(3)	4004(1)	35(1)
C(5)	2692(1)	-217(3)	3795(1)	36(1)
C(6)	2742(1)	-110(3)	3099(1)	36(1)
C(7)	1927(2)	1104(3)	4742(1)	43(1)
C(8)	1370(3)	-1356(6)	5499(2)	99(1)
C(9)	3321(1)	-2018(3)	4293(1)	44(1)
C(10)	4212(3)	-2990(5)	5500(2)	100(1)
C(11)	3500(1)	-1594(3)	2885(1)	44(1)
C(12)	5295(2)	-2323(5)	3171(2)	73(1)

Table 3. Bond lengths [A] and angles [deg] for 1.

Cl(1)-C(1)	1.720(2)
Cl(2)-C(2)	1.709(2)
Cl(3)-C(3)	1.722(2)
O(1)-C(7)	1.321(2)
O(1)-C(8)	1.454(3)
O(2)-C(7)	1.195(2)
O(3)-C(9)	1.322(2)
O(3)-C(10)	1.453(3)
O(4)-C(9)	1.195(2)
O(5)-C(11)	1.327(2)
O(5)-C(12)	1.447(2)
O(6)-C(11)	1.189(2)
C(1)-C(6)	1.385(2)
C(1)-C(2)	1.394(2)
C(2)-C(3)	1.394(2)
C(3)-C(4)	1.381(2)
C(4)-C(5)	1.399(2)
C(4)-C(7)	1.509(2)
C(5)-C(6)	1.394(2)
C(5)-C(9)	1.497(2)
C(6)-C(11)	1.508(2)

C(7)-O(1)-C(8)	116.6(2)
C(9)-O(3)-C(10)	116.0(2)
C(11)-O(5)-C(12)	116.7(2)
C(6)-C(1)-C(2)	120.2(2)
C(6)-C(1)-Cl(1)	120.38(12)
C(2)-C(1)-Cl(1)	119.38(13)
C(3)-C(2)-C(1)	119.17(14)
C(3)-C(2)-Cl(2)	120.67(12)
C(1)-C(2)-Cl(2)	120.16(12)
C(4)-C(3)-C(2)	121.26(14)
C(4)-C(3)-Cl(3)	119.86(12)
C(2)-C(3)-Cl(3)	118.83(12)
C(3)-C(4)-C(5)	119.1(2)
C(3)-C(4)-C(7)	119.73(14)
C(5)-C(4)-C(7)	121.1(2)
C(6)-C(5)-C(4)	120.2(2)
C(6)-C(5)-C(9)	118.1(2)
C(4)-C(5)-C(9)	121.6(2)
C(1)-C(6)-C(5)	120.0(2)
C(1)-C(6)-C(11)	119.4(2)
C(5)-C(6)-C(11)	120.4(2)
O(2)-C(7)-O(1)	125.6(2)
O(2)-C(7)-C(4)	124.9(2)
O(1)-C(7)-C(4)	109.49(14)
O(4)-C(9)-O(3)	124.9(2)
O(4)-C(9)-C(5)	123.2(2)
O(3)-C(9)-C(5)	111.9(2)
O(6)-C(11)-O(5)	125.0(2)
O(6)-C(11)-C(6)	126.1(2)
O(5)-C(11)-C(6)	108.88(14)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 1.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Cl(1)	49(1)	68(1)	35(1)	-1(1)	22(1)	-2(1)
Cl(2)	54(1)	63(1)	50(1)	18(1)	18(1)	19(1)
Cl(3)	60(1)	55(1)	54(1)	-6(1)	28(1)	16(1)
O(1)	102(1)	59(1)	43(1)	-7(1)	34(1)	-28(1)
O(2)	92(1)	56(1)	39(1)	-14(1)	26(1)	-14(1)
O(3)	89(1)	56(1)	43(1)	-2(1)	-10(1)	19(1)
O(4)	79(1)	44(1)	58(1)	4(1)	20(1)	14(1)
O(5)	34(1)	55(1)	70(1)	-6(1)	21(1)	6(1)
O(6)	62(1)	81(1)	80(1)	-39(1)	19(1)	10(1)
C(1)	32(1)	43(1)	32(1)	-4(1)	13(1)	-5(1)
C(2)	30(1)	38(1)	36(1)	2(1)	10(1)	0(1)
C(3)	32(1)	35(1)	38(1)	-6(1)	14(1)	-3(1)
C(4)	37(1)	38(1)	31(1)	-5(1)	12(1)	-5(1)
C(5)	36(1)	37(1)	35(1)	-3(1)	9(1)	-2(1)
C(6)	32(1)	39(1)	38(1)	-6(1)	13(1)	-2(1)
C(7)	52(1)	44(1)	33(1)	-3(1)	16(1)	-2(1)
C(8)	167(3)	90(2)	57(1)	0(1)	59(2)	-43(2)
C(9)	44(1)	43(1)	43(1)	2(1)	11(1)	0(1)
C(10)	135(3)	72(2)	54(1)	8(1)	-22(2)	32(2)
C(11)	42(1)	47(1)	46(1)	-4(1)	19(1)	5(1)
C(12)	50(1)	80(2)	98(2)	4(1)	36(1)	23(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 1.

	x	y	z	U(eq)
H(8A)	1023(3)	-2803(6)	5478(2)	149
H(8B)	978(3)	-167(6)	5630(2)	149
H(8C)	2051(3)	-1439(6)	5852(2)	149
H(10A)	4433(3)	-2297(5)	5969(2)	150
H(10B)	4807(3)	-3539(5)	5390(2)	150
H(10C)	3755(3)	-4252(5)	5493(2)	150
H(12A)	5949(2)	-1710(5)	3467(2)	110
H(12B)	5260(2)	-2280(5)	2675(2)	110
H(12C)	5229(2)	-3883(5)	3309(2)	110

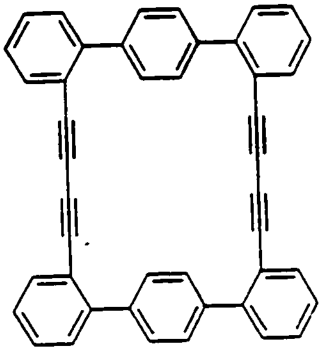


Table 1. Crystal data and structure refinement for af004a.

Identification code	af004a
Empirical formula	C45 H26 Cl2
Formula weight	637.56
Temperature	203(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 15.201(5) A alpha = 90 deg. b = 20.611(6) A beta = 90 deg. c = 21.317(6) A gamma = 90 deg.
Volume	6679(3) A^3
Z, Calculated density	8, 1.268 Mg/m^3
Absorption coefficient	0.226 mm^-1
F(000)	2640
Crystal size	0.3 x 0.04 x 0.04 mm
Theta range for data collection	1.98 to 20.99 deg.
Limiting indices	0<=h<=15, 0<=k<=20, 0<=l<=21
Reflections collected / unique	53136 / 3586 [R(int) = 0.7080]
Completeness to theta = 20.99	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3586 / 0 / 353
Goodness-of-fit on F^2	1.027
Final R indices [I>2sigma(I)]	R1 = 0.1125, wR2 = 0.2685
R indices (all data)	R1 = 0.2349, wR2 = 0.3704
Extinction coefficient	0.0015(6)
Largest diff. peak and hole	0.407 and -0.664 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af004a. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2577(5)	4884(4)	5264(3)	42(3)
C(2)	1890(6)	5329(4)	5287(4)	63(4)
C(3)	1295(5)	5313(4)	5782(5)	67(4)
C(4)	1387(5)	4852(5)	6254(4)	66(4)
C(5)	2074(6)	4408(4)	6231(3)	52(4)
C(6)	2669(5)	4424(4)	5736(4)	42(3)
C(7)	3358(5)	3894(4)	5719(4)	41(3)
C(8)	3117(4)	3244(4)	5740(4)	41(3)
C(9)	3755(5)	2764(3)	5692(4)	47(3)
C(10)	4635(5)	2933(4)	5624(4)	40(3)
C(11)	4876(4)	3584(4)	5604(4)	52(4)
C(12)	4238(6)	4064(3)	5651(4)	46(3)
C(13)	5346(4)	2409(3)	5577(4)	39(3)
C(14)	6080(5)	2477(3)	5961(3)	51(4)
C(15)	6762(4)	2030(4)	5926(4)	48(4)
C(16)	6711(4)	1514(4)	5507(4)	50(4)
C(17)	5977(6)	1446(3)	5123(4)	55(4)
C(18)	5295(4)	1894(4)	5158(3)	39(3)
C(19)	4604(8)	1817(6)	4705(6)	42(3)
C(20)	4076(9)	1758(6)	4284(7)	47(4)
C(21)	3481(8)	1717(6)	3795(7)	40(3)
C(22)	2981(8)	1707(6)	3364(6)	43(3)
C(23)	2314(4)	1733(4)	2891(3)	39(3)
C(24)	1637(6)	1279(3)	2914(4)	50(4)
C(25)	938(4)	1322(4)	2496(4)	54(4)
C(26)	915(4)	1817(4)	2054(4)	56(4)
C(27)	1592(6)	2271(4)	2031(3)	45(3)
C(28)	2291(4)	2228(3)	2449(4)	38(3)
C(29)	3006(4)	2741(3)	2408(4)	40(3)
C(30)	2782(4)	3393(4)	2460(4)	41(3)
C(31)	3432(5)	3866(3)	2426(4)	42(3)
C(32)	4306(4)	3687(3)	2340(4)	41(3)
C(33)	4529(3)	3035(4)	2288(3)	39(3)
C(34)	3879(5)	2562(3)	2322(3)	42(3)
C(35)	5039(4)	4202(4)	2307(4)	42(3)
C(36)	5623(5)	4165(3)	1807(3)	47(4)
C(37)	6321(5)	4599(4)	1766(4)	57(4)
C(38)	6434(4)	5070(4)	2226(4)	54(4)
C(39)	5849(6)	5107(3)	2726(4)	51(4)
C(40)	5152(5)	4673(4)	2767(3)	43(3)
C(41)	4582(8)	4726(6)	3285(7)	46(4)
C(42)	4115(8)	4807(6)	3736(7)	41(3)
C(43)	3595(8)	4858(6)	4274(7)	42(3)
C(44)	3142(9)	4891(6)	4730(7)	45(4)
C(45)	1088(10)	1252(8)	5866(10)	111(7)
Cl(1)	1833(4)	1707(3)	5445(3)	141(2)
Cl(2)	167(4)	1733(3)	6061(3)	136(2)

. Table 3. Bond lengths [A] and angles [deg] for af004a.

C(1)-C(2)	1.3900
C(1)-C(6)	1.3900
C(1)-C(44)	1.428(15)
C(2)-C(3)	1.3900
C(3)-C(4)	1.3900
C(4)-C(5)	1.3900
C(5)-C(6)	1.3900
C(6)-C(7)	1.514(8)
C(7)-C(8)	1.3900
C(7)-C(12)	1.3900
C(8)-C(9)	1.3900
C(9)-C(10)	1.3900
C(10)-C(11)	1.3900
C(10)-C(13)	1.532(8)
C(11)-C(12)	1.3900
C(13)-C(14)	1.3900
C(13)-C(18)	1.3900
C(14)-C(15)	1.3900
C(15)-C(16)	1.3900
C(16)-C(17)	1.3900
C(17)-C(18)	1.3900
C(18)-C(19)	1.437(15)
C(19)-C(20)	1.208(17)
C(20)-C(21)	1.38(2)
C(21)-C(22)	1.192(16)
C(22)-C(23)	1.432(15)
C(23)-C(24)	1.3900
C(23)-C(28)	1.3900
C(24)-C(25)	1.3900
C(25)-C(26)	1.3900
C(26)-C(27)	1.3900
C(27)-C(28)	1.3900
C(28)-C(29)	1.519(8)
C(29)-C(30)	1.3900
C(29)-C(34)	1.3900
C(30)-C(31)	1.3900
C(31)-C(32)	1.3900
C(32)-C(33)	1.3900
C(32)-C(35)	1.541(8)
C(33)-C(34)	1.3900
C(35)-C(36)	1.3900
C(35)-C(40)	1.3900
C(36)-C(37)	1.3900
C(37)-C(38)	1.3900
C(38)-C(39)	1.3900
C(39)-C(40)	1.3900
C(40)-C(41)	1.409(15)
C(41)-C(42)	1.206(17)
C(42)-C(43)	1.40(2)
C(43)-C(44)	1.192(17)
C(45)-Cl(1)	1.722(16)
C(45)-Cl(2)	1.765(16)

C(2)-C(1)-C(6)	120.0
C(2)-C(1)-C(44)	118.3(8)
C(6)-C(1)-C(44)	121.6(8)

C(3)-C(2)-C(1)	120.0
C(4)-C(3)-C(2)	120.0
C(3)-C(4)-C(5)	120.0
C(4)-C(5)-C(6)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-C(7)	116.8(7)
C(1)-C(6)-C(7)	123.0(7)
C(8)-C(7)-C(12)	120.0
C(8)-C(7)-C(6)	120.8(7)
C(12)-C(7)-C(6)	119.1(7)
C(7)-C(8)-C(9)	120.0
C(8)-C(9)-C(10)	120.0
C(11)-C(10)-C(9)	120.0
C(11)-C(10)-C(13)	119.4(6)
C(9)-C(10)-C(13)	120.6(6)
C(10)-C(11)-C(12)	120.0
C(11)-C(12)-C(7)	120.0
C(14)-C(13)-C(18)	120.0
C(14)-C(13)-C(10)	117.1(6)
C(18)-C(13)-C(10)	122.8(6)
C(13)-C(14)-C(15)	120.0
C(16)-C(15)-C(14)	120.0
C(17)-C(16)-C(15)	120.0
C(16)-C(17)-C(18)	120.0
C(17)-C(18)-C(13)	120.0
C(17)-C(18)-C(19)	115.9(7)
C(13)-C(18)-C(19)	123.9(7)
C(20)-C(19)-C(18)	174.4(13)
C(19)-C(20)-C(21)	177.7(14)
C(22)-C(21)-C(20)	177.1(14)
C(21)-C(22)-C(23)	173.6(13)
C(24)-C(23)-C(28)	120.0
C(24)-C(23)-C(22)	118.3(8)
C(28)-C(23)-C(22)	121.5(8)
C(23)-C(24)-C(25)	120.0
C(24)-C(25)-C(26)	120.0
C(27)-C(26)-C(25)	120.0
C(26)-C(27)-C(28)	120.0
C(27)-C(28)-C(23)	120.0
C(27)-C(28)-C(29)	117.8(7)
C(23)-C(28)-C(29)	122.2(7)
C(30)-C(29)-C(34)	120.0
C(30)-C(29)-C(28)	119.6(6)
C(34)-C(29)-C(28)	120.4(6)
C(29)-C(30)-C(31)	120.0
C(30)-C(31)-C(32)	120.0
C(31)-C(32)-C(33)	120.0
C(31)-C(32)-C(35)	120.9(6)
C(33)-C(32)-C(35)	119.1(6)
C(34)-C(33)-C(32)	120.0
C(33)-C(34)-C(29)	120.0
C(36)-C(35)-C(40)	120.0
C(36)-C(35)-C(32)	117.3(7)
C(40)-C(35)-C(32)	122.6(7)
C(37)-C(36)-C(35)	120.0
C(36)-C(37)-C(38)	120.0
C(39)-C(38)-C(37)	120.0
C(38)-C(39)-C(40)	120.0
C(39)-C(40)-C(35)	120.0
C(39)-C(40)-C(41)	117.9(8)

C(35)-C(40)-C(41)	122.1(8)
C(42)-C(41)-C(40)	176.1(13)
C(41)-C(42)-C(43)	175.9(14)
C(44)-C(43)-C(42)	178.7(14)
C(43)-C(44)-C(1)	175.9(13)
Cl(1)-C(45)-Cl(2)	109.8(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af004a.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	42(8)	42(9)	42(9)	-4(7)	-5(7)	0(7)
C(2)	85(11)	49(10)	56(11)	5(8)	9(9)	14(9)
C(3)	81(11)	47(10)	72(11)	-13(9)	13(10)	23(8)
C(4)	68(10)	62(11)	69(11)	1(9)	22(8)	10(9)
C(5)	62(9)	50(9)	43(9)	-1(7)	5(8)	-8(8)
C(6)	50(9)	43(9)	34(8)	-9(7)	-2(7)	-2(7)
C(7)	32(8)	58(10)	34(8)	-1(7)	2(6)	7(7)
C(8)	50(8)	40(9)	33(8)	-3(6)	-3(6)	2(8)
C(9)	40(8)	50(9)	52(9)	-3(7)	-2(7)	4(7)
C(10)	53(9)	48(9)	18(7)	7(6)	-7(6)	11(7)
C(11)	58(9)	42(9)	54(9)	-10(7)	-8(7)	-15(8)
C(12)	66(10)	22(7)	48(9)	1(6)	-1(7)	1(7)
C(13)	38(8)	35(8)	44(8)	1(7)	-4(7)	-6(6)
C(14)	57(9)	40(9)	55(9)	-14(7)	2(8)	1(8)
C(15)	43(8)	53(9)	48(9)	-1(8)	-8(7)	-6(7)
C(16)	46(9)	50(10)	54(9)	7(8)	10(7)	7(7)
C(17)	59(9)	60(11)	45(9)	-2(7)	-4(8)	3(8)
C(18)	30(7)	33(8)	56(9)	3(7)	1(7)	1(7)
C(19)	46(9)	28(8)	53(10)	3(7)	9(8)	-5(7)
C(20)	45(9)	35(8)	60(10)	-2(7)	3(8)	0(7)
C(21)	48(9)	28(8)	45(9)	-4(7)	2(8)	-3(6)
C(22)	48(9)	34(8)	47(9)	7(7)	4(8)	-4(7)
C(23)	40(8)	36(8)	39(8)	-10(7)	4(7)	13(7)
C(24)	37(8)	47(9)	65(10)	-12(7)	-1(7)	-2(7)
C(25)	36(8)	51(9)	75(11)	-22(9)	12(8)	-7(7)
C(26)	42(9)	65(10)	60(10)	-18(9)	-7(7)	4(8)
C(27)	51(9)	43(9)	41(9)	-3(7)	0(7)	2(7)
C(28)	39(8)	46(8)	31(8)	0(7)	3(6)	-3(6)
C(29)	44(8)	51(9)	24(8)	10(6)	4(6)	-4(7)
C(30)	30(7)	40(8)	53(9)	-3(7)	3(6)	6(7)
C(31)	51(8)	31(8)	45(9)	4(7)	3(7)	7(7)
C(32)	55(9)	41(9)	27(8)	6(6)	7(6)	-4(7)
C(33)	42(8)	45(9)	30(8)	-3(6)	7(6)	-4(7)
C(34)	44(8)	37(8)	44(9)	3(6)	-1(6)	1(7)
C(35)	34(8)	49(9)	43(9)	11(7)	-3(7)	4(6)
C(36)	45(8)	38(8)	58(10)	6(7)	6(7)	9(7)
C(37)	44(8)	41(9)	86(11)	9(8)	11(8)	-11(7)
C(38)	43(9)	33(9)	87(12)	5(8)	3(8)	-14(6)
C(39)	52(9)	42(9)	59(11)	5(7)	10(8)	-2(7)
C(40)	49(9)	43(8)	39(9)	-1(7)	-4(7)	2(7)
C(41)	54(9)	30(8)	53(10)	-3(7)	-1(8)	-7(7)
C(42)	47(8)	24(8)	53(10)	-3(7)	-3(8)	-10(6)
C(43)	44(8)	38(9)	44(9)	5(7)	-6(8)	-7(6)
C(44)	51(9)	31(8)	54(10)	-1(7)	1(8)	0(7)
C(45)	89(13)	60(11)	180(20)	12(13)	35(13)	5(10)
Cl(1)	133(5)	110(4)	180(6)	3(4)	-14(4)	-10(4)
Cl(2)	179(6)	106(4)	122(5)	1(4)	2(4)	-22(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af004a.

	x	y	z	U(eq)
H(2A)	1828	5640	4968	76
H(3A)	830	5613	5798	80
H(4A)	984	4841	6589	80
H(5A)	2136	4096	6550	62
H(8A)	2522	3129	5786	49
H(9A)	3592	2324	5706	57
H(11A)	5471	3698	5558	62
H(12A)	4401	4503	5638	55
H(14A)	6114	2826	6244	61
H(15A)	7258	2076	6186	58
H(16A)	7172	1211	5484	60
H(17A)	5943	1097	4840	66
H(24A)	1653	944	3213	60
H(25A)	481	1015	2512	65
H(26A)	443	1846	1771	67
H(27A)	1576	2606	1732	54
H(30A)	2192	3514	2518	49
H(31A)	3281	4307	2461	51
H(33A)	5120	2914	2229	47
H(34A)	4030	2122	2287	50
H(36A)	5547	3847	1496	57
H(37A)	6716	4574	1428	69
H(38A)	6906	5364	2199	65
H(39A)	5926	5426	3037	61
H(45A)	1366	1090	6250	133
H(45B)	900	878	5616	133

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
-10	6	10	71	64	15
-9	6	10	70	96	14
-8	6	10	145	144	9
-7	6	10	98	103	12
-6	6	10	85	83	11
-5	6	10	0	10	1
-4	6	10	23	3	22
-3	6	10	0	19	1
-2	6	10	27	1	27
-1	6	10	204	196	9
0	6	10	34	20	33
1	6	10	38	55	38
2	6	10	64	71	21
3	6	10	27	29	26
4	6	10	85	87	15
5	6	10	68	67	20
6	6	10	0	38	1
-13	7	10	85	46	20
-12	7	10	20	14	19
-11	7	10	61	49	27
-10	7	10	58	51	22
-9	7	10	40	30	40
-8	7	10	69	70	20
-7	7	10	98	95	10
-6	7	10	0	1	1
-5	7	10	49	54	48
-4	7	10	36	24	36
-3	7	10	152	139	10
-2	7	10	0	0	1
-1	7	10	52	56	28
0	7	10	59	51	29
1	7	10	90	99	16
2	7	10	0	15	1
3	7	10	9	42	9
4	7	10	0	12	1
5	7	10	30	8	30
-13	8	10	57	59	56
-12	8	10	34	0	33
-11	8	10	72	80	34
-10	8	10	167	155	13
-9	8	10	150	135	13
-8	8	10	168	159	10
-7	8	10	38	39	37
-6	8	10	35	1	34
-5	8	10	33	43	33
-4	8	10	0	16	1
-3	8	10	7	12	6
-2	8	10	18	2	18
-1	8	10	68	83	23
0	8	10	0	7	1
1	8	10	63	71	25
2	8	10	40	39	39
3	8	10	35	26	35
4	8	10	27	16	26
-11	9	10	44	46	44
-10	9	10	0	23	1
-9	9	10	88	76	26
-6	9	10	30	16	29
-5	9	10	0	1	1
-4	9	10	16	4	16
-3	9	10	13	2	12
-2	9	10	61	53	33
-1	9	10	0	11	1
0	9	10	0	21	1
1	9	10	51	56	51
2	9	10	40	49	40
-3	10	10	40	10	39
-2	10	10	61	47	40
-1	10	10	5	28	4
0	10	10	44	38	44
1	10	10	23	28	22
-1	11	10	52	14	52
-16	1	11	37	2	31
-15	1	11	28	6	27
-14	1	11	0	7	1
-13	1	11	25	26	25
-12	1	11	21	30	21
-11	1	11	56	62	10
-10	1	11	14	15	13
-9	1	11	0	13	1
-8	1	11	68	61	9
-7	1	11	42	39	15
-6	1	11	87	90	8
-5	1	11	96	107	8
-4	1	11	11	2	10
-3	1	11	103	104	11
-2	1	11	149	150	9
-1	1	11	30	25	30
0	1	11	206	186	6
1	1	11	29	8	29
2	1	11	284	255	6
3	1	11	73	81	17
4	1	11	82	70	18
5	1	11	0	10	1
6	1	11	46	11	45
-16	2	11	0	15	1
-15	2	11	76	82	8
-14	2	11	5	11	4
-13	2	11	0	8	1
-12	2	11	0	15	1
-11	2	11	46	50	13
-10	2	11	60	66	9
-9	2	11	105	113	5
-8	2	11	31	31	31
-7	2	11	54	65	13
-6	2	11	113	113	7
-5	2	11	45	35	19
-4	2	11	29	10	28
-3	2	11	12	3	11
-2	2	11	50	56	22
-1	2	11	243	238	7
0	2	11	72	66	15
1	2	11	72	60	14
2	2	11	133	114	9
3	2	11	29	11	29
4	2	11	40	45	40
5	2	11	35	33	35
6	2	11	67	68	29
7	2	11	0	32	1
-15	3	11	56	57	16
-14	3	11	62	65	12
-13	3	11	28	20	27
-12	3	11	20	37	20
-11	3	11	84	97	8
-10	3	11	53	52	12
-9	3	11	127	139	6
-8	3	11	119	127	6
-7	3	11	65	68	11
-6	3	11	142	137	7
-5	3	11	151	165	6
-4	3	11	182	184	6
-3	3	11	154	147	7
-2	3	11	80	73	15
-1	3	11	144	132	9
0	3	11	156	153	9
1	3	11	102	100	13
2	3	11	259	237	9
3	3	11	80	66	19
4	3	11	22	23	21
5	3	11	0	12	1
6	3	11	32	22	31
7	3	11	0	26	1
-15	4	11	60	70	18
-14	4	11	24	34	23
-13	4	11	26	39	25
-12	4	11	26	20	25
-11	4	11	28	38	27
-10	4	11	58	57	14
-9	4	11	116	123	7
-8	4	11	84	100	9
-7	4	11	70	72	11
-6	4	11	65	77	11
-5	4	11	32	31	32
-4	4	11	39	42	31
-3	4	11	50	67	18
-2	4	11	32	42	32
-1	4	11	18	0	18
0	4	11	128	125	11
1	4	11	39	46	39
2	4	11	49	44	49
3	4	11	17	6	17
4	4	11	0	29	1
5	4	11	25	12	25
6	4	11	44	56	43
-15	5	11	0	36	1
-14	5	11	29	30	29
-13	5	11	14	13	14
-12	5	11	22	15	22
-11	5	11	0	8	1
-10	5	11	20	24	19
-9	5	11	53	42	20
-8	5	11	0	6	1
-7	5	11	0	1	1
-6	5	11	195	186	8
-5	5	11	68	59	15
-4	5	11	147	135	7
-3	5	11	88	85	14
-2	5	11	0	11	1
-1	5	11	78	65	19
0	5	11	51	37	30
1	5	11	62	52	26
2	5	11	9	42	9
3	5	11	34	36	34
4	5	11	28	9	27
5	5	11	69	54	24
-14	6	11	0	11	1
-13	6	11	0	10	1
-12	6	11	40	33	40
-11	6	11	0	23	1
-10	6	11	27	14	27
-9	6	11	85	85	12
-8	6	11	56	65	24
-7	6	11	0	15	1
-6	6	11	107	90	10
-5	6	11	41	44	33
-4	6	11	63	58	22
-3	6	11	88	84	16
-2	6	11	21	23	21
-1	6	11	55	71	30
0	6	11	62	61	25
1	6	11	136	142	15
2	6	11	26	41	25
3	6	11	53	63	35
4	6	11	66	63	23
-13	7	11	28	2	27
-12	7	11	22	7	21
-11	7	11	78	61	17
-10	7	11	60	63	27
-9	7	11	65	66	22
-8	7	11	74	71	20
-7	7	11	47	51	32
-6	7	11	0	5	1
-5	7	11	113	95	14
-4	7	11	29	41	28
-3	7	11	43	51	42
-2	7	11	60	61	30
-1	7	11	0	11	1
0	7	11	74	56	23
1	7	11	25	2	24
2	7	11	49	63	48
3	7	11	0	20	1
-12	8	11	5	18	4
-11	8	11	78	75	36
-10	8	11	64	46	43
-9	8	11	14	5	14
-8	8	11	50	26	50
-7	8	11	39	11	38
-6	8	11	134	129	13
-5	8	11	135	131	14
-4	8	11	0	17	1
-3	8	11	0	2	1
-2	8	11	93	88	16
-1	8	11	0	0	1
0	8	11	0	21	1
1	8	11	0	4	1
2	8	11	22	19	21
-11	9	11	33	0	33
-10	9	11	125	110	20
-7	9	11	0	2	1
-6	9	11	55	42	54
-5	9	11	28	40	28
-4	9	11	56	50	55
-3	9	11	48	43	48
-2	9	11	0	25	1
-1	9	11	0	32	1
0	9	11	0	4	1
1	9	11	27	6	26
-4	10	11	127	109	23
-3	10	11	0	6	1
-2	10	11	69	42	37
-1	10	11	0	7	1
-16	0	12	86	53	23
-15	0	12	18	20	18
-14	0	12	0	10	1
-13	0	12	51	54	17
-12	0	12	57	72	13
-11	0	12	110	103	9
-10	0	12	50	51	21
-9	0	12	86	96	10
-8	0	12	132	134	7
-7	0	12	184	186	7
-6	0	12	151	165	11
-5	0	12	185	195	11
-4	0	12	0	32	1
-3	0	12	37	22	36
-2	0	12	0	24	1
-1	0	12	47	57	46
0	0	12	343	306	9
1	0	12	79	118	22
2	0	12	78	77	23
3	0	12	80	70	22
-16	1	12	82	81	25
-15	1	12	40	56	16
-14	1	12	18	30	18
-13	1	12	0	3	1
-12	1	12	94	100	6
-11	1	12	137	153	5
-10	1	12	53	67	10
-9	1	12	14	11	13
-8	1	12	60	64	10
-7	1	12	80	89	9
-6	1	12	13	28	13
-5	1	12	32	34	31
-4	1	12	74	85	14
-3	1	12	0	12	1
-2	1	12	103	105	13
-1	1	12	22	15	22
0	1	12	28	12	28
1	1	12	142	139	7
2	1	12	61	66	25
3	1	12	33	31	33
4	1	12	71	69	25
5	1	12	99	83	18
-15	2	12	29	18	28
-14	2	12	22	16	22
-13	2	12	61	64	10
-12	2	12	91	92	7
-11	2	12	25	33	25
-10	2	12	119	122	5
-9	2	12	24	19	24
-8	2	12	7	20	6
-7	2	12	21	21	20
-6	2	12	0	7	1
-5	2	12	74	76	10
-4	2	12	16	18	15
-3	2	12	111	120	11
-2	2	12	12	18	12
-1	2	12	109	103	10
0	2	12	49	50	35
1	2	12	75	65	15
2	2	12	36	43	35
3	2	12	0	18	1
4	2	12	21	18	20
5	2	12	0	26	1
6	2	12	24	21	24
-15	3	12	17	20	17
-14	3	12	55	61	14
-13	3	12	0	4	1
-12	3	12	35	27	34
-11	3	12	34	47	24
-10	3	12	39	50	23
-9	3	12	90	99	8
-8	3	12	181	198	7
-7	3	12	185	180	6
-6	3	12	34	38	34
-5	3	12	240	250	6
-4	3	12	114	121	11
-3	3	12	0	10	1
-2	3	12	231	218	14
-1	3	12	0	18	1
0	3	12	131	120	12
1	3	12	19	31	18
2	3	12	121	111	13
3	3	12	0	18	1
4	3	12	51	45	50
5	3	12	0	1	1
6	3	12	78	71	28
-15	4	12	0	0	1
-14	4	12	25	25	25
-13	4	12	53	46	15
-12	4	12	42	49	28
-11	4	12	93	96	9
-10	4	12	44	37	22
-9	4	12	0	17	1
-8	4	12	167	165	6
-7	4	12	100	101	8
-6	4	12	103	111	8
-5	4	12	0	5	1
-4	4	12	51	42	20
-3	4	12	67	85	19
-2	4	12	43	42	43
-1	4	12	34	59	33
0	4	12	56	51	30
1	4	12	146	125	12
2	4	12	0	17	1
3	4	12	0	10	1
4	4	12	83	71	19
5	4	12	37	10	37
-14	5	12	36	39	35
-13	5	12	27	0	26
-12	5	12	25	17	24
-11	5	12	104	96	10
-10	5	12	8	2	8
-9	5	12	19	5	18
-8	5	12	9	42	9
-7	5	12	89	102	11
-6	5	12	78	72	17
-5	5	12	112	115	9
-4	5	12	0	28	1
-3	5	12	28	42	28
-2	5	12	107	107	14
-1	5	12	121	92	13
0	5	12	0	2	1
1	5	12	38	20	38
2	5	12	32	50	31
3	5	12	136	122	12
4	5	12	39	38	38
-14	6	12	23	24	23
-13	6	12	0	22	1
-12	6	12	0	11	1
-11	6	12	0	12	1
-10	6	12	26	18	25
-9	6	12	72	74	17
-8	6	12	0	34	1
-7	6	12	55	55	24
-6	6	12	38	6	38
-5	6	12	63	68	26
-4	6	12	23	10	23
-3	6	12	39	19	38
-2	6	12	51	38	44
-1	6	12	68	60	21
0	6	12	59	37	32
1	6	12	133	110	13
2	6	12	87	94	19
3	6	12	26	34	26
-13	7	12	49	1	48
-12	7	12	30	29	30
-11	7	12	72	46	25
-10	7	12	52	63	48
-9	7	12	21	3	21
-8	7	12	0	9	1
-7	7	12	65	48	19
-6	7	12	48	32	48
-5	7	12	0	8	1
-4	7	12	31	51	31
-3	7	12	0	23	1
-2	7	12	0	10	1
-1	7	12	0	19	1
0	7	12	35	18	34
1	7	12	0	3	1
2	7	12	27	1	27
-11	8	12	0	1	1
-10	8	12	172	144	11
-9	8	12	64	64	30
-8	8	12	0	81	1
-7	8	12	25	4	25
-6	8	12	37	27	37
-5	8	12	0	15	1
-4	8	12	34	27	33
-3	8	12	0	8	1
-2	8	12	25	24	25
-1	8	12	58	64	50
0	8	12	66	56	30
1	8	12	82	67	42
-7	9	12	0	10	1
-6	9	12	15	52	14
-5	9	12	40	17	40
-4	9	12	0	21	1
-3	9	12	69	51	33
-2	9	12	0	3	1
-1	9	12	8	5	8
-5	10	12	75	86	75
-4	10	12	0	22	1
-3	10	12	0	5	1
-15	1	13	12	5	11

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
-14	1	13	17	17	17
-13	1	13	51	53	12
-12	1	13	25	29	25
-11	1	13	59	69	10
-10	1	13	12	21	11
-9	1	13	78	84	8
-8	1	13	53	60	14
-7	1	13	105	111	8
-6	1	13	58	65	18
-5	1	13	44	59	30
-4	1	13	0	29	1
-3	1	13	0	18	1
-2	1	13	181	176	7
-1	1	13	32	20	31
0	1	13	89	83	13
1	1	13	81	68	19
2	1	13	0	11	1
3	1	13	25	41	25
4	1	13	0	6	1
-15	2	13	34	15	34
-14	2	13	54	55	13
-13	2	13	31	32	31
-12	2	13	37	43	22
-11	2	13	56	52	11
-10	2	13	98	108	7
-9	2	13	25	28	25
-8	2	13	104	109	8
-7	2	13	142	148	6
-6	2	13	91	85	9
-5	2	13	48	59	28
-4	2	13	34	46	34
-3	2	13	7	3	6
-2	2	13	21	8	20
-1	2	13	298	275	9
0	2	13	39	50	39
1	2	13	72	62	24
2	2	13	35	11	35
3	2	13	0	18	1
4	2	13	11	3	11
5	2	13	39	53	39
-15	3	13	0	5	1
-14	3	13	19	46	18
-13	3	13	31	44	31
-12	3	13	17	19	17
-11	3	13	15	6	15
-10	3	13	25	9	25
-9	3	13	74	86	11
-8	3	13	0	2	1
-7	3	13	67	68	13
-6	3	13	81	80	10
-5	3	13	100	103	9
-4	3	13	35	39	34
-3	3	13	8	6	7
-2	3	13	111	108	14
-1	3	13	30	19	30
0	3	13	52	41	51
1	3	13	49	56	48
2	3	13	55	49	54
3	3	13	0	1	1
4	3	13	40	44	40
-14	4	13	5	4	4
-13	4	13	0	13	1
-12	4	13	0	2	1
-11	4	13	95	108	9
-10	4	13	37	30	36
-9	4	13	43	36	24
-8	4	13	25	34	25
-7	4	13	11	17	11
-6	4	13	67	79	17
-5	4	13	81	75	13
-4	4	13	52	45	32
-3	4	13	30	20	29
-2	4	13	15	22	15
-1	4	13	84	84	20
0	4	13	127	122	13
1	4	13	45	43	44
2	4	13	0	3	1
3	4	13	46	26	46
4	4	13	0	14	1
-14	5	13	13	12	13
-13	5	13	12	37	11
-12	5	13	54	53	20
-11	5	13	41	43	36
-10	5	13	58	61	17
-9	5	13	30	12	29
-8	5	13	0	51	1
-7	5	13	17	2	16
-6	5	13	19	40	18
-5	5	13	84	90	12
-4	5	13	34	13	34
-3	5	13	57	49	33
-2	5	13	48	24	47
-1	5	13	57	60	37
0	5	13	80	74	22
1	5	13	68	67	29
2	5	13	21	11	20
3	5	13	0	0	1
-13	6	13	0	14	1
-12	6	13	63	59	20
-11	6	13	75	87	19
-10	6	13	71	70	19
-9	6	13	0	22	1
-8	6	13	82	62	18
-7	6	13	68	56	18
-6	6	13	41	21	41
-5	6	13	43	6	43
-4	6	13	96	102	19
-3	6	13	26	40	25
-2	6	13	35	32	35
-1	6	13	78	83	21
0	6	13	27	19	27
1	6	13	43	24	42
-12	7	13	0	9	1
-11	7	13	25	37	25
-10	7	13	55	63	33
-9	7	13	0	2	1
-8	7	13	42	44	41
-7	7	13	0	22	1
-6	7	13	0	11	1
-5	7	13	21	37	21
-4	7	13	64	62	31
-3	7	13	77	89	25
-2	7	13	0	18	1
-1	7	13	23	40	23
0	7	13	44	23	44
-10	8	13	75	68	26
-9	8	13	21	39	20
-8	8	13	51	45	51
-7	8	13	52	50	52
-6	8	13	41	5	40
-5	8	13	0	20	1
-4	8	13	0	2	1
-3	8	13	14	8	13
-2	8	13	69	55	31
-1	8	13	0	5	1
-8	9	13	32	40	32
-7	9	13	0	28	1
-6	9	13	16	19	15
-5	9	13	33	1	32
-4	9	13	49	31	48
-3	9	13	52	39	52
-14	0	14	0	7	1
-13	0	14	54	48	20
-12	0	14	80	103	11
-11	0	14	0	9	1
-10	0	14	25	45	25
-9	0	14	33	24	32
-8	0	14	0	7	1
-7	0	14	0	11	1
-6	0	14	76	74	22
-5	0	14	34	52	33
-4	0	14	118	122	18
-3	0	14	18	54	18
-2	0	14	89	77	18
-1	0	14	136	114	14
0	0	14	42	45	42
1	0	14	0	4	1
2	0	14	80	48	56
-14	1	14	43	42	18
-13	1	14	10	2	9
-12	1	14	81	85	8
-11	1	14	128	149	5
-10	1	14	31	39	31
-9	1	14	3	7	3
-8	1	14	163	171	6
-7	1	14	167	181	8
-6	1	14	88	106	13
-5	1	14	35	42	34
-4	1	14	20	20	20
-3	1	14	136	143	11
-2	1	14	53	38	29
-1	1	14	107	100	13
0	1	14	172	152	9
1	1	14	20	20	19
2	1	14	32	4	31
3	1	14	81	59	55
-14	2	14	0	3	1
-13	2	14	0	0	1
-12	2	14	0	20	1
-11	2	14	44	59	17
-10	2	14	33	30	32
-9	2	14	57	76	14
-8	2	14	58	71	14
-7	2	14	30	43	30
-6	2	14	60	69	19
-5	2	14	37	48	37
-4	2	14	0	20	1
-3	2	14	169	165	10
-2	2	14	78	54	22
-1	2	14	38	32	38
0	2	14	60	57	33
1	2	14	31	3	30
2	2	14	0	0	1
3	2	14	0	8	1
4	2	14	30	32	30
-14	3	14	0	10	1
-13	3	14	13	20	13
-12	3	14	28	30	27
-11	3	14	0	21	1
-10	3	14	42	46	32
-9	3	14	89	102	9
-8	3	14	30	39	29
-7	3	14	92	105	10
-6	3	14	58	57	16
-5	3	14	43	56	43
-4	3	14	111	116	13
-3	3	14	0	7	1
-2	3	14	115	103	15
-1	3	14	102	108	18
0	3	14	0	16	1
1	3	14	0	25	1
2	3	14	49	41	49
3	3	14	0	12	1
-13	4	14	0	21	1
-12	4	14	58	69	18
-11	4	14	63	58	16
-10	4	14	0	7	1
-9	4	14	0	1	1
-8	4	14	49	57	21
-7	4	14	66	96	17
-6	4	14	9	0	9
-5	4	14	16	15	15
-4	4	14	101	99	14
-3	4	14	103	101	16
-2	4	14	49	31	49
-1	4	14	33	31	33
0	4	14	109	109	16
1	4	14	31	5	31
2	4	14	118	88	18
-13	5	14	44	39	43
-12	5	14	0	12	1
-11	5	14	6	8	5
-10	5	14	61	63	21
-9	5	14	20	35	20
-8	5	14	16	29	16
-7	5	14	46	44	45
-6	5	14	0	23	1
-5	5	14	52	27	51
-4	5	14	47	14	46
-3	5	14	63	62	34
-2	5	14	33	20	33
-1	5	14	52	55	51
0	5	14	56	66	55
1	5	14	33	1	33
-12	6	14	0	2	1
-11	6	14	0	13	1
-10	6	14	31	19	31
-9	6	14	29	15	28
-8	6	14	46	45	46
-7	6	14	0	10	1
-6	6	14	0	8	1
-5	6	14	25	7	25
-4	6	14	17	24	16
-3	6	14	78	85	23
-2	6	14	136	109	14
-1	6	14	61	73	39
-10	7	14	8	11	8
-9	7	14	0	16	1
-8	7	14	39	32	39
-7	7	14	47	34	47
-6	7	14	58	45	52
-5	7	14	0	24	1
-4	7	14	29	31	28
-3	7	14	59	39	58
-2	7	14	67	53	31
-7	8	14	95	73	26
-6	8	14	26	21	26
-5	8	14	47	24	47
-4	8	14	0	15	1
-13	1	15	0	7	1
-12	1	15	0	4	1
-11	1	15	77	88	8
-10	1	15	0	7	1
-9	1	15	0	18	1
-8	1	15	54	71	24
-7	1	15	39	54	39
-6	1	15	21	18	21
-5	1	15	28	44	28
-4	1	15	85	74	18
-3	1	15	0	8	1
-2	1	15	47	58	47
-1	1	15	69	61	25
0	1	15	29	17	29
1	1	15	38	7	37
2	1	15	76	63	76
-13	2	15	76	85	15
-12	2	15	0	11	1
-11	2	15	0	4	1
-10	2	15	134	148	7
-9	2	15	27	27	27
-8	2	15	41	43	29
-7	2	15	37	50	37
-6	2	15	0	37	1
-5	2	15	77	88	17
-4	2	15	0	19	1
-3	2	15	49	11	48
-2	2	15	30	42	29
-1	2	15	18	11	18
0	2	15	46	38	46
1	2	15	64	58	64
2	2	15	0	32	1
-13	3	15	21	32	20
-12	3	15	0	7	1
-11	3	15	34	46	34
-10	3	15	0	28	1
-9	3	15	0	3	1
-8	3	15	0	17	1
-7	3	15	28	35	27
-6	3	15	25	29	25
-5	3	15	33	44	33
-4	3	15	32	13	32
-3	3	15	0	27	1
-2	3	15	98	89	21
-1	3	15	0	13	1
0	3	15	113	99	18
1	3	15	56	5	56
-13	4	15	23	20	23
-12	4	15	43	30	42
-11	4	15	25	21	25
-10	4	15	24	14	23
-9	4	15	13	23	13
-8	4	15	43	26	42
-7	4	15	0	14	1
-6	4	15	85	75	18
-5	4	15	0	24	1
-4	4	15	82	82	21
-3	4	15	30	4	29
-2	4	15	40	34	39
-1	4	15	30	23	29
0	4	15	12	30	12
-12	5	15	14	17	14
-11	5	15	30	40	29
-10	5	15	0	7	1
-9	5	15	27	21	27
-8	5	15	94	99	14
-7	5	15	64	49	19
-6	5	15	32	18	31
-5	5	15	78	97	21
-4	5	15	49	58	49
-3	5	15	28	29	27
-2	5	15	0	4	1
-10	6	15	39	23	39
-9	6	15	43	40	43
-8	6	15	58	58	26
-7	6	15	42	42	41
-6	6	15	8	12	8
-5	6	15	0	13	1
-4	6	15	0	27	1
-9	7	15	0	10	[illegible]
-8	7	15	83	49	[illegible]
-7	7	15	116	82	[illegible]
-6	7	15	0	6	
-12	0	16	98	116	17
-11	0	16	0	18	1
-10	0	16	161	180	13
-9	0	16	30	42	30
-8	0	16	28	11	28
-7	0	16	0	6	1
-6	0	16	103	122	20
-5	0	16	59	57	58
-4	0	16	74	58	38
-3	0	16	0	38	1
-2	0	16	0	6	1
-1	0	16	39	41	38
0	0	16	0	8	1
-12	1	16	38	56	38
-11	1	16	0	33	1
-10	1	16	113	121	8
-9	1	16	92	105	13
-8	1	16	92	122	14
-7	1	16	20	24	19
-6	1	16	46	29	46
-5	1	16	0	20	1
-4	1	16	17	30	17
-3	1	16	28	22	27
-2	1	16	60	52	36
-1	1	16	0	20	1
0	1	16	0	20	1
-12	2	16	27	18	27
-11	2	16	0	19	1
-10	2	16	22	46	22
-9	2	16	38	44	38
-8	2	16	26	30	25
-7	2	16	0	4	1
-6	2	16	0	25	1
-5	2	16	33	22	32
-4	2	16	60	59	35
-3	2	16	86	89	22
-2	2	16	53	45	52
-1	2	16	81	62	30
0	2	16	29	0	28
-12	3	16	0	3	1
-11	3	16	0	26	1
-10	3	16	0	25	1
-9	3	16	0	7	1
-8	3	16	30	45	29
-7	3	16	15	30	15
-6	3	16	44	26	43
-5	3	16	30	27	29
-4	3	16	95	84	21
-3	3	16	93	82	21
-2	3	16	49	25	49
-1	3	16	14	19	13
-12	4	16	0	10	1
-11	4	16	22	41	22
-10	4	16	60	63	48
-9	4	16	26	23	25
-8	4	16	10	8	10
-7	4	16	0	15	1
-6	4	16	29	14	28
-5	4	16	0	10	1
-4	4	16	168	133	19
-3	4	16	96	94	32
-9	5	16	30	39	29
-8	5	16	56	28	48
-7	5	16	73	75	53
-6	5	16	0	7	1
-9	6	16	48	45	48
-10	1	17	37	45	36
-9	1	17	0	20	1
-8	1	17	18	5	17
-7	1	17	22	14	21
-6	1	17	27	5	27
-5	1	17	14	30	14
-4	1	17	83	58	25
-3	1	17	52	59	52
-2	1	17	0	16	1
-10	2	17	48	49	47
-9	2	17	68	68	37
-8	2	17	22	21	21
-7	2	17	40	31	40
-6	2	17	34	50	33
-5	2	17	0	9	1
-4	2	17	37	41	36
-3	2	17	27	38	26

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
-10	3	17	79	67	33
-7	1	18	56	89	56

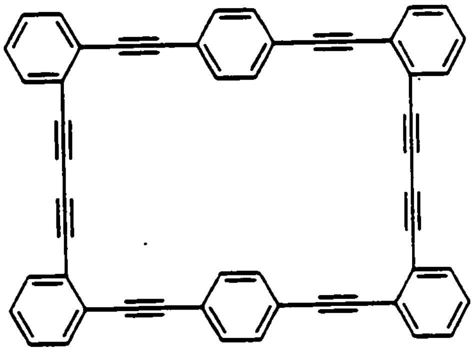


Table 1. Crystal data and structure refinement for af005a.

Identification code	af005a
Empirical formula	C52 H24
Formula weight	648.71
Temperature	203(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbcn
Unit cell dimensions	a = 30.657(3) A alpha = 90 deg. b = 8.5590(7) A beta = 90 deg. c = 13.7917(12) A gamma = 90 deg.
Volume	3618.9(5) A^3
Z, Calculated density	4, 1.191 Mg/m^3
Absorption coefficient	0.068 mm^-1
F(000)	1344
Crystal size	0.1 x 0.3 x 0.4 mm
Theta range for data collection	1.33 to 28.74 deg.
Limiting indices	0<=h<=41, 0<=k<=11, 0<=l<=17
Reflections collected / unique	28287 / 4437 [R(int) = 0.0883]
Completeness to theta = 28.74	94.4 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4437 / 0 / 236
Goodness-of-fit on F^2	1.077
Final R indices [I>2sigma(I)]	R1 = 0.0504, wR2 = 0.1105
R indices (all data)	R1 = 0.0960, wR2 = 0.1203
Extinction coefficient	0.0079(7)
Largest diff. peak and hole	0.215 and -0.262 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af005a. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	1761(1)	4678(2)	7767(1)	45(1)
C(2)	1773(1)	3411(2)	7394(1)	47(1)
C(3)	1779(1)	1972(2)	6951(1)	48(1)
C(4)	1778(1)	703(2)	6578(1)	48(1)
C(5)	1759(1)	-823(2)	6166(1)	44(1)
C(6)	2138(1)	-1672(2)	5987(1)	52(1)
C(7)	2115(1)	-3149(2)	5590(1)	61(1)
C(8)	1715(1)	-3820(2)	5396(1)	64(1)
C(9)	1337(1)	-3000(2)	5571(1)	58(1)
C(10)	1351(1)	-1491(2)	5944(1)	46(1)
C(11)	966(1)	-567(2)	6050(1)	53(1)
C(12)	664(1)	314(2)	6092(1)	57(1)
C(13)	322(1)	1456(2)	6133(1)	56(1)
C(14)	-112(1)	1019(2)	6229(1)	63(1)
C(15)	-433(1)	2148(2)	6303(1)	63(1)
C(16)	-327(1)	3722(2)	6279(1)	56(1)
C(17)	106(1)	4158(2)	6166(1)	63(1)
C(18)	428(1)	3030(2)	6101(1)	64(1)
C(19)	-666(1)	4870(2)	6373(1)	58(1)
C(20)	-963(1)	5772(2)	6440(1)	53(1)
C(21)	-1335(1)	6779(2)	6466(1)	46(1)
C(22)	-1314(1)	8301(2)	6121(1)	58(1)
C(23)	-1679(1)	9235(2)	6097(1)	60(1)
C(24)	-2074(1)	8644(2)	6399(1)	58(1)
C(25)	-2105(1)	7149(2)	6765(1)	50(1)
C(26)	-1736(1)	6201(2)	6813(1)	44(1)

Table 3. Bond lengths [A] and angles [deg] for af005a.

C(1)-C(2)	1.201(2)
C(1)-C(26)#1	1.428(2)
C(2)-C(3)	1.375(2)
C(3)-C(4)	1.202(2)
C(4)-C(5)	1.426(2)
C(5)-C(6)	1.391(2)
C(5)-C(10)	1.4081(19)
C(6)-C(7)	1.380(2)
C(7)-C(8)	1.381(2)
C(8)-C(9)	1.375(2)
C(9)-C(10)	1.391(2)
C(10)-C(11)	1.428(2)
C(11)-C(12)	1.197(2)
C(12)-C(13)	1.434(2)
C(13)-C(18)	1.387(2)
C(13)-C(14)	1.387(2)
C(14)-C(15)	1.383(2)
C(15)-C(16)	1.387(2)
C(16)-C(17)	1.386(2)
C(16)-C(19)	1.437(2)
C(17)-C(18)	1.385(2)
C(19)-C(20)	1.197(2)
C(20)-C(21)	1.430(2)
C(21)-C(22)	1.389(2)
C(21)-C(26)	1.407(2)
C(22)-C(23)	1.375(2)
C(23)-C(24)	1.377(2)
C(24)-C(25)	1.380(2)
C(25)-C(26)	1.395(2)
C(26)-C(1)#1	1.428(2)

C(2)-C(1)-C(26)#1	178.04(19)
C(1)-C(2)-C(3)	178.6(2)
C(4)-C(3)-C(2)	178.6(2)
C(3)-C(4)-C(5)	177.09(19)
C(6)-C(5)-C(10)	119.36(14)
C(6)-C(5)-C(4)	120.95(15)
C(10)-C(5)-C(4)	119.68(14)
C(7)-C(6)-C(5)	120.39(16)
C(6)-C(7)-C(8)	120.25(17)
C(9)-C(8)-C(7)	120.07(16)
C(8)-C(9)-C(10)	120.85(16)
C(9)-C(10)-C(5)	119.03(15)
C(9)-C(10)-C(11)	121.73(15)
C(5)-C(10)-C(11)	119.13(14)
C(12)-C(11)-C(10)	173.86(19)
C(11)-C(12)-C(13)	176.09(19)
C(18)-C(13)-C(14)	119.37(17)
C(18)-C(13)-C(12)	119.25(16)
C(14)-C(13)-C(12)	121.36(18)
C(15)-C(14)-C(13)	120.07(18)
C(14)-C(15)-C(16)	120.63(17)
C(17)-C(16)-C(15)	119.28(16)
C(17)-C(16)-C(19)	121.22(18)
C(15)-C(16)-C(19)	119.49(15)
C(18)-C(17)-C(16)	120.18(18)

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
2	0	0	137	217	18
4	0	0	1227	1129	27
6	0	0	1124	1239	45
8	0	0	190	305	78
10	0	0	188	172	87
12	0	0	349	247	50
14	0	0	0	229	1
2	1	0	574	818	5
4	1	0	368	400	11
6	1	0	154	158	33
8	1	0	106	144	85
10	1	0	0	116	1
12	1	0	308	301	41
14	1	0	608	513	38
0	2	0	1490	1749	18
2	2	0	482	569	10
4	2	0	934	1004	7
6	2	0	335	419	14
8	2	0	338	395	18
10	2	0	342	329	21
12	2	0	358	344	44
14	2	0	632	587	27
2	3	0	214	146	15
4	3	0	591	552	9
6	3	0	380	392	14
8	3	0	360	334	17
10	3	0	277	278	28
12	3	0	75	262	74
14	3	0	484	489	34
0	4	0	1276	945	8
2	4	0	628	577	9
4	4	0	709	718	8
6	4	0	191	193	29
8	4	0	1064	913	16
10	4	0	236	285	38
12	4	0	709	699	20
14	4	0	45	170	44
2	5	0	1406	1483	8
4	5	0	0	85	1
6	5	0	403	269	17
8	5	0	430	432	17
10	5	0	241	281	53
12	5	0	402	321	37
14	5	0	98	12	98
0	6	0	4674	4456	18
2	6	0	548	443	9
4	6	0	316	382	17
6	6	0	611	699	10
8	6	0	337	295	30
10	6	0	314	113	42
12	6	0	0	172	1
14	6	0	57	117	56
2	7	0	831	720	7
4	7	0	609	611	10
6	7	0	375	292	22
8	7	0	366	349	29
10	7	0	0	78	1
12	7	0	208	142	91
14	7	0	0	115	1
0	8	0	87	93	87
2	8	0	0	160	1
4	8	0	2412	2353	10
6	8	0	376	393	18
8	8	0	480	463	22
10	8	0	104	149	104
12	8	0	269	297	66
14	8	0	122	10	121
2	9	0	0	84	1
4	9	0	290	272	26
6	9	0	402	357	19
8	9	0	106	163	105
10	9	0	108	57	107
12	9	0	0	88	1
0	10	0	0	28	1
2	10	0	530	502	16
4	10	0	113	124	113
6	10	0	0	46	1
8	10	0	276	316	40
10	10	0	554	592	27
12	10	0	125	214	125
2	11	0	1133	1051	10
4	11	0	378	346	22
6	11	0	412	456	29
8	11	0	316	371	44
10	11	0	175	222	117
12	11	0	0	14	1
0	12	0	1064	1056	16
2	12	0	249	190	43
4	12	0	252	240	39
6	12	0	130	55	129
8	12	0	130	26	129
10	12	0	396	269	40
12	12	0	0	81	1
2	13	0	1001	963	14
4	13	0	590	464	22
6	13	0	432	425	26
8	13	0	347	338	37
10	13	0	283	375	62
0	14	0	980	987	21
2	14	0	302	275	42
4	14	0	0	97	1
6	14	0	325	272	42
8	14	0	123	33	123
10	14	0	137	31	137
2	15	0	224	270	66
4	15	0	259	283	81
6	15	0	213	9	78
8	15	0	154	182	153
10	15	0	273	269	71
0	16	0	214	209	93
2	16	0	169	143	168
4	16	0	0	109	1
6	16	0	80	143	80
8	16	0	0	35	1
2	17	0	300	8	59
4	17	0	143	82	143
6	17	0	189	83	111
8	17	0	0	54	1
0	18	0	0	68	1
2	18	0	1093	1162	18
4	18	0	0	9	1
6	18	0	254	234	93
2	19	0	485	508	42
4	19	0	0	178	1
6	19	0	336	178	93
0	20	0	715	658	35
2	20	0	0	55	1
4	20	0	277	5	101
2	1	1	238	166	7
3	1	1	543	516	4
4	1	1	270	298	12
5	1	1	566	606	6
6	1	1	127	56	30
7	1	1	113	104	38
8	1	1	288	302	14
9	1	1	0	5	1
10	1	1	0	78	1
11	1	1	153	147	50
12	1	1	149	155	79
13	1	1	178	182	81
14	1	1	0	35	1
15	1	1	171	121	171
0	2	1	555	740	5
1	2	1	629	415	3
2	2	1	1651	1733	5
3	2	1	234	177	10
4	2	1	666	751	5
5	2	1	433	434	7
6	2	1	620	653	5
7	2	1	0	97	1
8	2	1	0	0	1
9	2	1	0	56	1
10	2	1	139	139	42
11	2	1	159	140	49
12	2	1	136	188	135
13	2	1	171	166	88
14	2	1	245	161	60
15	2	1	266	26	74
1	3	1	247	242	9
2	3	1	309	183	9
3	3	1	264	259	14
4	3	1	547	508	7
5	3	1	934	1110	6
6	3	1	0	105	1
7	3	1	763	718	7
8	3	1	692	633	9
9	3	1	169	192	32
10	3	1	60	82	60
11	3	1	143	89	55
12	3	1	539	466	17
13	3	1	137	122	137
14	3	1	272	360	45
15	3	1	416	336	49
0	4	1	1427	1203	5
1	4	1	1248	1366	4
2	4	1	604	438	6
3	4	1	0	52	1
4	4	1	606	688	6
5	4	1	531	515	6
6	4	1	45	150	44
7	4	1	161	44	27
8	4	1	511	434	11
9	4	1	134	121	51
10	4	1	150	21	51
11	4	1	220	245	41
12	4	1	143	220	123
13	4	1	205	187	60
14	4	1	112	112	111
15	4	1	0	126	1
1	5	1	422	235	9
2	5	1	327	287	11
3	5	1	156	212	22
4	5	1	596	593	7
5	5	1	551	661	8
6	5	1	351	275	13
7	5	1	171	191	23
8	5	1	540	468	10
9	5	1	242	238	28
10	5	1	184	230	49
11	5	1	169	61	70
12	5	1	254	236	40
13	5	1	0	60	1
14	5	1	229	73	56
0	6	1	323	390	21
1	6	1	741	703	7
2	6	1	1111	1126	4
3	6	1	45	79	44
4	6	1	102	177	45
5	6	1	425	376	8
6	6	1	847	832	9
7	6	1	180	129	29
8	6	1	78	57	77
9	6	1	354	355	21
10	6	1	127	154	126
11	6	1	87	127	87
12	6	1	0	43	1
13	6	1	0	75	1
14	6	1	130	164	129
1	7	1	599	607	8
2	7	1	211	174	19
3	7	1	106	17	49
4	7	1	807	733	6
5	7	1	970	960	6
6	7	1	161	157	27
7	7	1	20	21	20
8	7	1	87	4	87
9	7	1	131	113	86
10	7	1	171	139	63
11	7	1	215	153	58
12	7	1	0	100	1
13	7	1	0	40	1
14	7	1	158	65	157
0	8	1	298	375	27
1	8	1	645	688	8
2	8	1	171	166	28
3	8	1	330	314	13
4	8	1	197	184	23
5	8	1	117	69	47
6	8	1	0	81	1
7	8	1	577	609	11
8	8	1	203	248	39
9	8	1	0	11	1
10	8	1	229	211	44
11	8	1	149	80	113
12	8	1	0	101	1
13	8	1	0	66	1
14	8	1	0	35	1
1	9	1	644	493	9
2	9	1	889	889	6
3	9	1	627	602	9
4	9	1	655	618	9
5	9	1	770	805	7
6	9	1	120	133	56
7	9	1	0	31	1
8	9	1	450	417	16
9	9	1	136	41	82
10	9	1	131	132	131
11	9	1	0	41	1
12	9	1	0	101	1
13	9	1	0	22	1
0	10	1	450	373	22
1	10	1	1001	987	6
2	10	1	268	297	23
3	10	1	560	470	10
4	10	1	219	234	25
5	10	1	155	22	38
6	10	1	372	342	17
7	10	1	260	335	29
8	10	1	224	224	36
9	10	1	186	276	48
10	10	1	104	159	104
11	10	1	0	22	1
12	10	1	244	245	52
13	10	1	75	94	74
1	11	1	144	105	54
2	11	1	894	840	9
3	11	1	866	788	8
4	11	1	281	241	21
5	11	1	472	427	15
6	11	1	344	367	21
7	11	1	0	127	1
8	11	1	137	22	80
9	11	1	0	138	1
10	11	1	324	383	31
11	11	1	229	281	52
12	11	1	0	47	1
0	12	1	288	209	43
1	12	1	134	122	77
2	12	1	314	239	23
3	12	1	0	3	1
4	12	1	263	246	27
5	12	1	40	7	40
6	12	1	333	296	25
7	12	1	0	5	1
8	12	1	90	120	89
9	12	1	156	68	78
10	12	1	212	189	54
11	12	1	185	171	78
12	12	1	0	24	1
1	13	1	80	136	80
2	13	1	104	185	104
3	13	1	0	56	1
4	13	1	619	550	13
5	13	1	150	109	67
6	13	1	139	29	78
7	13	1	20	12	20
8	13	1	212	234	46
9	13	1	150	131	89
10	13	1	165	255	85
11	13	1	125	88	125
0	14	1	60	156	60
1	14	1	0	84	1
2	14	1	192	155	50
3	14	1	535	474	17
4	14	1	367	324	25
5	14	1	53	49	52
6	14	1	66	12	66
7	14	1	259	253	36
8	14	1	72	19	72
9	14	1	100	39	100
10	14	1	288	290	42
11	14	1	215	192	70
1	15	1	0	35	1
2	15	1	78	153	77
3	15	1	0	83	1
4	15	1	182	220	84
5	15	1	234	111	43
6	15	1	169	34	72
7	15	1	110	131	109
8	15	1	193	157	58
9	15	1	215	118	63
10	15	1	243	197	56
0	16	1	171	221	96
1	16	1	672	608	16
2	16	1	0	109	1
3	16	1	244	207	54
4	16	1	202	180	82
5	16	1	0	76	1
6	16	1	0	133	1
7	16	1	227	164	50
8	16	1	221	253	51
9	16	1	0	1	1
1	17	1	165	131	85
2	17	1	123	16	123
3	17	1	588	571	24
4	17	1	60	101	60
5	17	1	341	208	44
6	17	1	0	3	1
7	17	1	0	92	1
8	17	1	0	62	1
0	18	1	278	342	57
1	18	1	0	40	1
2	18	1	335	335	39
3	18	1	268	272	57
4	18	1	140	99	140
5	18	1	0	13	1
6	18	1	85	61	84
7	18	1	0	52	1
1	19	1	194	241	59
2	19	1	349	318	42
3	19	1	144	132	144
4	19	1	0	101	1
5	19	1	96	12	96
6	19	1	231	141	82
0	20	1	401	307	41
1	20	1	186	253	77
2	20	1	98	138	98
3	20	1	0	198	1
4	20	1	90	31	89
2	0	2	249	313	11
3	0	2	502	383	10
4	0	2	178	194	26
5	0	2	267	272	15
6	0	2	973	1014	6
7	0	2	0	22	1
8	0	2	321	364	18
9	0	2	57	101	56
10	0	2	0	49	1
11	0	2	235	235	43
12	0	2	113	154	113
13	0	2	53	39	52
14	0	2	868	729	28
15	0	2	226	1	153
2	1	2	464	586	4
3	1	2	247	367	13
4	1	2	410	460	7
5	1	2	230	339	15
6	1	2	506	541	7
7	1	2	0	91	1
8	1	2	0	59	1
9	1	2	139	163	38
10	1	2	78	73	77
11	1	2	154	162	47
12	1	2	398	343	19
13	1	2	0	146	1
14	1	2	395	341	37
15	1	2	100	113	100
0	2	2	155	318	17
1	2	2	576	515	3
2	2	2	3727	3331	8
3	2	2	978	887	5
4	2	2	179	176	21
5	2	2	143	127	27
6	2	2	1043	1033	6
7	2	2	470	478	9
8	2	2	63	5	63
9	2	2	63	130	63
10	2	2	0	33	1
11	2	2	0	31	1
12	2	2	45	54	44
13	2	2	151	126	131
14	2	2	0	58	1
15	2	2	0	40	1
1	3	2	92	75	31
2	3	2	840	851	5
3	3	2	83	15	82
4	3	2	176	150	24
5	3	2	192	222	17
6	3	2	526	500	7
7	3	2	57	68	56
8	3	2	120	106	50
9	3	2	453	459	12
10	3	2	0	64	1
11	3	2	136	96	63
12	3	2	335	330	28
13	3	2	569	501	21
14	3	2	147	81	147
15	3	2	0	35	1
0	4	2	407	442	17
1	4	2	119	159	28
2	4	2	556	588	8
3	4	2	836	859	6
4	4	2	451	429	9
5	4	2	321	303	13
6	4	2	113	195	38

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
7	4	2	169	159	28
8	4	2	0	63	1
9	4	2	154	120	38
10	4	2	368	345	19
11	4	2	248	178	36
12	4	2	0	22	1
13	4	2	0	167	1
14	4	2	0	2	1
1	5	2	1331	1510	5
2	5	2	949	1050	4
3	5	2	172	88	21
4	5	2	1101	1176	5
5	5	2	618	521	8
6	5	2	418	422	12
7	5	2	92	138	91
8	5	2	143	162	38
9	5	2	436	376	15
10	5	2	85	28	84
11	5	2	0	115	1
12	5	2	269	291	39
13	5	2	208	180	56
14	5	2	0	6	1
0	6	2	1601	1757	7
1	6	2	984	801	5
2	6	2	351	380	11
3	6	2	281	295	13
4	6	2	223	193	18
5	6	2	521	538	9
6	6	2	1369	1344	7
7	6	2	293	294	18
8	6	2	227	184	26
9	6	2	72	132	72
10	6	2	195	229	52
11	6	2	292	243	35
12	6	2	113	166	113
13	6	2	160	105	90
14	6	2	337	281	43
1	7	2	2220	2329	5
2	7	2	522	373	8
3	7	2	319	349	13
4	7	2	371	366	10
5	7	2	156	180	34
6	7	2	421	450	13
7	7	2	49	65	49
8	7	2	314	308	20
9	7	2	154	111	59
10	7	2	356	349	26
11	7	2	176	179	73
12	7	2	166	142	88
13	7	2	0	37	1
14	7	2	0	39	1
0	8	2	939	990	9
1	8	2	195	218	28
2	8	2	0	10	1
3	8	2	108	139	58
4	8	2	634	581	9
5	8	2	506	480	8
6	8	2	276	296	22
7	8	2	220	259	27
8	8	2	394	421	18
9	8	2	75	96	74
10	8	2	134	65	134
11	8	2	125	193	125
12	8	2	28	42	28
13	8	2	0	20	1
14	8	2	0	50	1
1	9	2	92	15	91
2	9	2	455	403	12
3	9	2	90	128	89
4	9	2	1018	1013	6
5	9	2	0	72	1
6	9	2	45	26	44
7	9	2	283	278	23
8	9	2	484	507	15
9	9	2	372	335	23
10	9	2	102	106	102
11	9	2	0	80	1
12	9	2	80	89	80
13	9	2	140	50	140
0	10	2	159	45	76
1	10	2	45	84	44
2	10	2	351	250	18
3	10	2	558	476	10
4	10	2	173	175	34
5	10	2	57	74	56
6	10	2	85	107	84
7	10	2	169	133	48
8	10	2	0	42	1
9	10	2	80	96	80
10	10	2	0	31	1
11	10	2	0	39	1
12	10	2	106	116	105
13	10	2	171	41	96
1	11	2	35	20	34
2	11	2	592	534	12
3	11	2	346	292	17
4	11	2	703	616	10
5	11	2	561	592	12
6	11	2	0	27	1
7	11	2	0	36	1
8	11	2	0	30	1
9	11	2	246	251	41
10	11	2	0	140	1
11	11	2	0	62	1
12	11	2	263	246	46
0	12	2	587	506	20
1	12	2	326	275	23
2	12	2	128	67	79
3	12	2	324	284	21
4	12	2	60	94	60
5	12	2	0	43	1
6	12	2	264	257	32
7	12	2	239	193	35
8	12	2	381	340	23
9	12	2	125	99	125
10	12	2	0	128	1
11	12	2	108	91	107
12	12	2	172	137	92
1	13	2	959	870	11
2	13	2	49	42	49
3	13	2	123	50	123
4	13	2	154	171	57
5	13	2	324	298	25
6	13	2	139	67	89
7	13	2	296	301	30
8	13	2	0	124	1
9	13	2	0	130	1
10	13	2	180	284	72
11	13	2	144	15	144
0	14	2	319	340	43
1	14	2	0	47	1
2	14	2	207	93	45
3	14	2	127	147	126
4	14	2	0	7	1
5	14	2	252	325	37
6	14	2	123	105	123
7	14	2	256	238	41
8	14	2	346	360	27
9	14	2	191	209	61
10	14	2	0	16	1
11	14	2	0	170	1
1	15	2	249	140	40
2	15	2	0	98	1
3	15	2	412	338	24
4	15	2	0	37	1
5	15	2	234	137	48
6	15	2	125	33	125
7	15	2	143	119	108
8	15	2	179	224	69
9	15	2	110	45	109
10	15	2	0	156	1
0	16	2	75	100	74
1	16	2	0	35	1
2	16	2	123	29	123
3	16	2	205	181	60
4	16	2	793	751	17
5	16	2	136	146	135
6	16	2	130	98	129
7	16	2	0	16	1
8	16	2	218	114	57
9	16	2	35	109	34
1	17	2	0	43	1
2	17	2	221	174	58
3	17	2	348	259	35
4	17	2	460	430	31
5	17	2	312	258	45
6	17	2	0	143	1
7	17	2	0	75	1
8	17	2	0	64	1
0	18	2	49	59	49
1	18	2	263	207	46
2	18	2	304	316	41
3	18	2	0	92	1
4	18	2	0	28	1
5	18	2	0	131	1
6	18	2	0	84	1
7	18	2	225	105	67
1	19	2	635	674	18
2	19	2	0	84	1
3	19	2	178	238	125
4	19	2	0	7	1
5	19	2	0	44	1
6	19	2	206	20	98
0	20	2	338	325	55
1	20	2	0	1	1
2	20	2	72	33	72
3	20	2	0	43	1
1	1	3	171	175	10
2	1	3	879	982	3
3	1	3	1436	1546	5
4	1	3	127	101	32
5	1	3	305	344	10
6	1	3	1069	1053	5
7	1	3	0	57	1
8	1	3	0	30	1
9	1	3	0	30	1
10	1	3	287	297	21
11	1	3	262	231	25
12	1	3	78	2	77
13	1	3	0	126	1
14	1	3	278	340	53
15	1	3	433	332	41
0	2	3	965	578	5
1	2	3	579	412	5
2	2	3	607	396	7
3	2	3	231	151	15
4	2	3	924	814	4
5	2	3	131	97	25
6	2	3	108	124	41
7	2	3	139	3	44
8	2	3	78	62	77
9	2	3	319	310	16
10	2	3	321	291	18
11	2	3	0	7	1
12	2	3	172	95	53
13	2	3	0	202	1
14	2	3	370	347	39
15	2	3	0	99	1
1	3	3	113	23	33
2	3	3	7887	6641	11
3	3	3	151	197	27
4	3	3	603	682	5
5	3	3	187	208	19
6	3	3	806	828	5
7	3	3	654	646	8
8	3	3	462	436	11
9	3	3	231	239	24
10	3	3	0	35	1
11	3	3	363	408	21
12	3	3	272	204	32
13	3	3	0	37	1
14	3	3	362	361	39
15	3	3	154	142	153
0	4	3	1095	1464	8
1	4	3	1116	975	4
2	4	3	399	275	10
3	4	3	404	492	8
4	4	3	1197	1285	5
5	4	3	422	453	10
6	4	3	582	666	6
7	4	3	294	245	15
8	4	3	200	193	24
9	4	3	262	253	24
10	4	3	0	105	1
11	4	3	285	248	30
12	4	3	629	571	17
13	4	3	215	228	53
14	4	3	0	11	1
1	5	3	504	552	8
2	5	3	646	586	6
3	5	3	2213	2312	4
4	5	3	149	260	28
5	5	3	146	143	32
6	5	3	1727	1608	7
7	5	3	401	386	13
8	5	3	69	6	69
9	5	3	20	61	20
10	5	3	411	399	21
11	5	3	0	35	1
12	5	3	281	248	36
13	5	3	130	4	129
14	5	3	227	174	62
0	6	3	208	176	37
1	6	3	1274	1277	4
2	6	3	405	325	10
3	6	3	820	774	6
4	6	3	349	294	12
5	6	3	636	652	7
6	6	3	432	406	13
7	6	3	0	20	1
8	6	3	218	194	29
9	6	3	315	294	22
10	6	3	271	211	32
11	6	3	215	157	52
12	6	3	330	378	31
13	6	3	0	48	1
14	6	3	240	202	52
1	7	3	764	687	6
2	7	3	746	673	5
3	7	3	100	100	55
4	7	3	0	33	1
5	7	3	396	379	12
6	7	3	69	109	69
7	7	3	332	287	16
8	7	3	228	187	27
9	7	3	189	19	50
10	7	3	328	273	29
11	7	3	209	106	56
12	7	3	263	171	51
13	7	3	150	94	115
14	7	3	104	186	104
0	8	3	1463	1485	9
1	8	3	96	26	96
2	8	3	517	476	9
3	8	3	707	607	8
4	8	3	937	846	6
5	8	3	20	109	20
6	8	3	231	292	23
7	8	3	87	44	87
8	8	3	302	244	24
9	8	3	90	48	89
10	8	3	147	142	90
11	8	3	210	173	56
12	8	3	0	12	1
13	8	3	0	81	1
1	9	3	346	338	17
2	9	3	102	178	102
3	9	3.	450	503	12
4	9	3	0	100	1
5	9	3	0	39	1
6	9	3	143	175	49
7	9	3	276	260	22
8	9	3	0	125	1
9	9	3	0	54	1
10	9	3	139	10	118
11	9	3	180	181	70
12	9	3	0	24	1
13	9	3	153	40	152
0	10	3	606	553	16
1	10	3	0	34	1
2	10	3	591	482	10
3	10	3	0	22	1
4	10	3	691	670	9
5	10	3	0	42	1
6	10	3	503	498	13
7	10	3	92	54	91
8	10	3	139	33	75
9	10	3	244	154	39
10	10	3	0	48	1
11	10	3	0	54	1
12	10	3	0	120	1
13	10	3	0	99	1
1	11	3	143	153	60
2	11	3	342	284	19
3	11	3	682	654	9
4	11	3	0	32	1
5	11	3	69	73	69
6	11	3	427	408	16
7	11	3	233	191	35
8	11	3	211	95	43
9	11	3	326	228	29
10	11	3	0	112	1
11	11	3	155	227	106
12	11	3	123	109	123
0	12	3	404	387	27
1	12	3	20	3	20
2	12	3	140	78	65
3	12	3	717	664	11
4	12	3	170	132	46
5	12	3	113	81	113
6	12	3	0	88	1
7	12	3	207	125	43
8	12	3	306	263	33
9	12	3	80	193	80
10	12	3	431	419	24
11	12	3	137	57	137
12	12	3	117	175	116
1	13	3	381	413	22
2	13	3	139	174	82
3	13	3	169	58	55
4	13	3	333	254	25
5	13	3	492	451	16
6	13	3	78	123	77
7	13	3	0	17	1
8	13	3	297	287	35
9	13	3	0	71	1
10	13	3	329	336	33
11	13	3	0	56	1
0	14	3	389	397	38
1	14	3	0	107	1
2	14	3	144	60	84
3	14	3	435	417	22
4	14	3	143	176	79
5	14	3	134	119	94
6	14	3	0	74	1
7	14	3	175	144	63
8	14	3	125	183	125
9	14	3	0	14	1
10	14	3	199	314	66
11	14	3	189	113	81
1	15	3	104	137	104
2	15	3	436	382	24
3	15	3	519	490	20
4	15	3	436	421	25
5	15	3	199	175	60
6	15	3	0	77	1
7	15	3	90	101	89
8	15	3	57	102	56
9	15	3	0	21	1
10	15	3	53	28	52
0	16	3	94	234	93
1	16	3	94	74	93
2	16	3	551	552	20
3	16	3	69	42	69
4	16	3	993	876	13
5	16	3	0	146	1
6	16	3	0	10	1
7	16	3	100	48	100
8	16	3	139	130	138
9	16	3	0	81	1
1	17	3	0	44	1
2	17	3	574	539	21
3	17	3	528	548	22
4	17	3	0	148	1
5	17	3	0	113	1
6	17	3	0	31	1
7	17	3	144	34	144
8	17	3	0	41	1
0	18	3	383	340	39
1	18	3	225	179	58
2	18	3	379	327	34
3	18	3	287	293	50
4	18	3	171	18	118
5	18	3	159	117	158
6	18	3	0	95	1
7	18	3	0	57	1
1	19	3	223	231	61
2	19	3	184	145	98
3	19	3	128	70	128
4	19	3	248	219	78
5	19	3	136	218	135
0	20	3	49	56	49
1	20	3	198	196	66
2	20	3	319	310	44
3	20	3	0	100	1
0	0	4	3318	3084	12
1	0	4	2074	2342	11
2	0	4	316	569	12
3	0	4	1062	1068	7
4	0	4	1085	954	7
5	0	4	128	35	50
6	0	4	475	411	12
7	0	4	338	332	15
8	0	4	416	424	15
9	0	4	365	358	21

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
10	0	4	102	102	102
11	0	4	187	50	77
12	0	4	253	259	49
13	0	4	180	288	105
14	0	4	144	128	144
15	0	4	288	155	106
1	1	4	921	779	3
2	1	4	253	229	11
3	1	4	549	434	8
4	1	4	484	548	7
5	1	4	412	244	9
6	1	4	352	274	12
7	1	4	365	276	12
8	1	4	0	89	1
9	1	4	92	99	91
10	1	4	216	155	31
11	1	4	0	28	1
12	1	4	125	30	125
13	1	4	200	164	54
14	1	4	352	394	39
15	1	4	0	131	1
0	2	4	908	790	8
1	2	4	1232	1177	4
2	2	4	261	377	12
3	2	4	1214	1236	4
4	2	4	203	239	18
5	2	4	473	465	8
6	2	4	882	849	5
7	2	4	125	182	44
8	2	4	169	154	30
9	2	4	220	264	27
10	2	4	113	142	93
11	2	4	234	212	36
12	2	4	488	478	18
13	2	4	0	74	1
14	2	4	0	8	1
1	3	4	856	902	6
2	3	4	214	196	21
3	3	4	347	389	10
4	3	4	847	861	5
5	3	4	370	420	10
6	3	4	365	321	11
7	3	4	469	421	11
8	3	4	113	152	60
9	3	4	190	164	32
10	3	4	0	8	1
11	3	4	134	108	77
12	3	4	433	399	19
13	3	4	169	149	76
14	3	4	0	7	1
0	4	4	267	296	26
1	4	4	988	1128	7
2	4	4	651	495	6
3	4	4	438	449	8
4	4	4	1088	1096	4
5	4	4	294	256	13
6	4	4	426	372	9
7	4	4	654	668	6
8	4	4	0	28	1
9	4	4	272	268	23
10	4	4	137	40	59
11	4	4	106	19	105
12	4	4	184	171	58
13	4	4	315	286	32
14	4	4	0	97	1
1	5	4	1079	1201	7
2	5	4	1636	1603	5
3	5	4	98	149	53
4	5	4	1175	1246	5
5	5	4	196	208	23
6	5	4	469	442	12
7	5	4	153	97	39
8	5	4	585	508	12
9	5	4	49	23	49
10	5	4	325	327	23
11	5	4	355	364	25
12	5	4	364	385	26
13	5	4	139	6	110
14	5	4	184	120	85
0	6	4	339	289	19
1	6	4	490	586	12
2	6	4	752	904	5
3	6	4	1420	1433	4
4	6	4	201	147	19
5	6	4	212	251	21
6	6	4	917	837	7
7	6	4	441	443	12
8	6	4	490	407	14
9	6	4	233	266	31
10	6	4	0	83	1
11	6	4	453	402	20
12	6	4	100	41	100
13	6	4	139	1	138
14	6	4	98	160	98
1	7	4	389	438	13
2	7	4	712	680	6
3	7	4	159	181	30
4	7	4	0	16	1
5	7	4	175	147	30
6	7	4	440	463	12
7	7	4	253	242	23
8	7	4	133	132	63
9	7	4	215	107	42
10	7	4	45	55	44
11	7	4	139	160	118
12	7	4	0	159	1
13	7	4	134	122	134
14	7	4	139	88	138
0	8	4	1273	1262	8
1	8	4	840	869	7
2	8	4	813	678	7
3	8	4	524	498	9
4	8	4	289	227	17
5	8	4	104	113	84
6	8	4	130	79	52
7	8	4	180	199	35
8	8	4	180	167	45
9	8	4	83	101	82
10	8	4	144	120	95
11	8	4	217	122	57
12	8	4	283	282	43
13	8	4	118	163	118
1	9	4	848	753	8
2	9	4	226	227	26
3	9	4	808	747	7
4	9	4	415	400	13
5	9	4	497	475	11
6	9	4	187	196	34
7	9	4	166	188	44
8	9	4	191	164	45
9	9	4	0	4	1
10	9	4	0	142	1
11	9	4	127	104	126
12	9	4	130	13	129
13	9	4	123	18	123
0	10	4	342	225	28
1	10	4	130	150	76
2	10	4	227	194	28
3	10	4	483	447	12
4	10	4	380	294	15
5	10	4	92	143	91
6	10	4	0	96	1
7	10	4	75	60	74
8	10	4	98	45	98
9	10	4	0	22	1
10	10	4	254	287	42
11	10	4	147	127	118
12	10	4	0	109	1
13	10	4	0	118	1
1	11	4	321	311	23
2	11	4	367	333	20
3	11	4	110	179	109
4	11	4	522	559	13
5	11	4	242	258	29
6	11	4	155	47	57
7	11	4	143	9	76
8	11	4	250	266	38
9	11	4	0	75	1
10	11	4	413	342	26
11	11	4	49	50	49
12	11	4	200	129	64
0	12	4	161	245	95
1	12	4	0	24	1
2	12	4	528	505	17
3	12	4	681	634	11
4	12	4	0	51	1
5	12	4	298	301	23
6	12	4	92	122	91
7	12	4	191	238	49
8	12	4	173	136	67
9	12	4	329	340	32
10	12	4	80	49	80
11	12	4	90	214	89
12	12	4	0	52	1
1	13	4	160	207	64
2	13	4	0	2	1
3	13	4	69	15	69
4	13	4	244	195	35
5	13	4	254	255	32
6	13	4	190	267	44
7	13	4	0	111	1
8	13	4	0	25	1
9	13	4	143	116	108
10	13	4	239	201	51
11	13	4	0	75	1
0	14	4	516	530	24
1	14	4	553	535	17
2	14	4	0	93	1
3	14	4	503	483	21
4	14	4	100	102	100
5	14	4	509	493	17
6	14	4	85	49	84
7	14	4	352	294	27
8	14	4	53	54	52
9	14	4	222	260	51
10	14	4	66	108	66
1	15	4	269	256	36
2	15	4	57	124	56
3	15	4	155	25	102
4	15	4	366	324	32
5	15	4	96	103	96
6	15	4	137	165	137
7	15	4	169	183	72
8	15	4	45	54	44
9	15	4	112	20	111
10	15	4	0	50	1
0	16	4	83	135	82
1	16	4	75	121	74
2	16	4	475	463	24
3	16	4	703	686	17
4	16	4	369	298	37
5	16	4	202	283	62
6	16	4	125	90	125
7	16	4	0	21	1
8	16	4	0	82	1
9	16	4	0	28	1
1	17	4	235	180	53
2	17	4	0	226	1
3	17	4	260	250	55
4	17	4	0	65	1
5	17	4	0	54	1
6	17	4	0	69	1
7	17	4	0	3	1
8	17	4	98	37	98
0	18	4	28	115	28
1	18	4	262	263	47
2	18	4	596	425	25
3	18	4	98	174	98
4	18	4	0	121	1
5	18	4	0	27	1
6	18	4	154	140	153
7	18	4	0	26	1
1	19	4	403	363	29
2	19	4	131	59	131
3	19	4	186	100	110
4	19	4	85	56	84
5	19	4	0	74	1
0	20	4	80	42	80
1	20	4	307	252	38
2	20	4	298	170	50
3	20	4	0	46	1
1	1	5	1318	1449	4
2	1	5	405	468	10
3	1	5	356	402	8
4	1	5	557	491	8
5	1	5	358	335	10
6	1	5	474	473	10
7	1	5	115	67	54
8	1	5	115	6	65
9	1	5	108	28	87
10	1	5	0	25	1
11	1	5	0	1	1
12	1	5	252	220	38
13	1	5	218	69	52
14	1	5	246	294	54
0	2	5	640	751	6
1	2	5	894	964	6
2	2	5	1461	1661	4
3	2	5	1643	1766	4
4	2	5	28	95	28
5	2	5	228	225	18
6	2	5	1086	1050	6
7	2	5	694	655	7
8	2	5	408	375	13
9	2	5	96	152	96
10	2	5	188	178	40
11	2	5	160	203	62
12	2	5	94	132	93
13	2	5	0	64	1
14	2	5	0	149	1
1	3	5	2277	2570	6
2	3	5	295	385	12
3	3	5	1661	1797	4
4	3	5	850	934	5
5	3	5	314	369	11
6	3	5	182	137	27
7	3	5	155	137	34
8	3	5	788	741	8
9	3	5	255	246	26
10	3	5	366	304	20
11	3	5	35	120	34
12	3	5	747	627	14
13	3	5	301	335	35
14	3	5	223	242	62
0	4	5	584	666	12
1	4	5	35	56	34
2	4	5	816	909	5
3	4	5	529	539	8
4	4	5	305	287	13
5	4	5	115	117	48
6	4	5	208	121	24
7	4	5	133	173	43
8	4	5	735	731	7
9	4	5	49	33	49
10	4	5	195	272	37
11	4	5	0	56	1
12	4	5	100	52	100
13	4	5	290	271	35
14	4	5	0	25	1
1	5	5	278	383	17
2	5	5	806	842	5
3	5	5	492	585	9
4	5	5	1175	1073	6
5	5	5	256	337	17
6	5	5	0	51	1
7	5	5	422	359	15
8	5	5	473	438	14
9	5	5	40	1	40
10	5	5	0	152	1
11	5	5	191	205	48
12	5	5	168	156	63
13	5	5	171	249	75
14	5	5	45	63	44
0	6	5	72	69	72
1	6	5	245	278	21
2	6	5	267	283	17
3	6	5	388	365	12
4	6	5	125	9	40
5	6	5	311	305	17
6	6	5	865	824	8
7	6	5	110	146	75
8	6	5	247	207	28
9	6	5	261	186	30
10	6	5	442	378	19
11	6	5	302	328	29
12	6	5	127	18	126
13	6	5	211	89	54
14	6	5	94	13	93
1	7	5	359	356	14
2	7	5	0	74	1
3	7	5	110	86	60
4	7	5	332	289	13
5	7	5	259	283	19
6	7	5	0	97	1
7	7	5	0	6	1
8	7	5	0	7	1
9	7	5	160	139	62
10	7	5	134	145	99
11	7	5	136	203	107
12	7	5	0	5	1
13	7	5	146	70	117
0	8	5	930	842	19
1	8	5	45	91	44
2	8	5	1018	991	6
3	8	5	745	705	7
4	8	5	311	328	17
5	8	5	0	47	1
6	8	5	248	232	24
7	8	5	182	135	38
8	8	5	275	269	24
9	8	5	179	102	54
10	8	5	151	112	84
11	8	5	0	31	1
12	8	5	57	83	56
13	8	5	0	76	1
1	9	5	147	187	49
2	9	5	170	201	37
3	9	5	761	774	8
4	9	5	698	641	10
5	9	5	460	437	14
6	9	5	484	487	14
7	9	5	92	136	91
8	9	5	0	32	1
9	9	5	66	59	66
10	9	5	302	250	32
11	9	5	186	199	62
12	9	5	168	219	84
13	9	5	186	175	76
0	10	5	324	379	28
1	10	5	0	118	1
2	10	5	316	353	19
3	10	5	929	876	8
4	10	5	225	223	27
5	10	5	172	180	39
6	10	5	122	75	87
7	10	5	248	244	32
8	10	5	0	156	1
9	10	5	176	90	62
10	10	5	0	32	1
11	10	5	117	51	116
12	10	5	0	69	1
1	11	5	535	540	14
2	11	5	747	700	11
3	11	5	904	866	8
4	11	5	0	51	1
5	11	5	413	388	17
6	11	5	139	52	66
7	11	5	134	165	81
8	11	5	228	290	42
9	11	5	146	179	96
10	11	5	231	264	48
11	11	5	45	24	44
12	11	5	0	25	1
0	12	5	311	236	47
1	12	5	166	50	54
2	12	5	193	220	47
3	12	5	123	56	95
4	12	5	113	78	113
5	12	5	345	357	22
6	12	5	382	426	22
7	12	5	0	138	1
8	12	5	403	385	26
9	12	5	0	5	1
10	12	5	131	117	131
11	12	5	245	228	49
1	13	5	0	58	1
2	13	5	285	267	32
3	13	5	125	111	125
4	13	5	63	22	63
5	13	5	0	5	1
6	13	5	411	418	21
7	13	5	0	192	1
8	13	5	221	160	47
9	13	5	0	35	1
10	13	5	100	104	100
11	13	5	63	57	63
0	14	5	267	232	52
1	14	5	188	217	55
2	14	5	500	445	19
3	14	5	110	132	109
4	14	5	87	52	87
5	14	5	209	185	51
6	14	5	571	556	18
7	14	5	364	367	26
8	14	5	0	94	1
9	14	5	0	12	1
10	14	5	92	155	91
1	15	5	0	78	1
2	15	5	20	19	20
3	15	5	325	357	34
4	15	5	330	446	36
5	15	5	283	217	38
6	15	5	108	55	107
7	15	5	140	47	140
8	15	5	0	149	1

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
9	15	5	0	114	1
0	16	5	239	230	75
1	16	5	168	101	73
2	16	5	187	30	71
3	16	5	364	345	33
4	16	5	63	84	63
5	16	5	251	323	46
6	16	5	0	53	1
7	16	5	0	83	1
8	16	5	151	94	122
9	16	5	163	195	93
1	17	5	151	78	111
2	17	5	0	31	1
3	17	5	155	8	155
4	17	5	104	38	104
5	17	5	253	200	61
6	17	5	0	15	1
7	17	5	256	253	56
8	17	5	0	59	1
0	18	5	102	52	102
1	18	5	131	93	131
2	18	5	112	136	111
3	18	5	155	14	155
4	18	5	0	63	1
5	18	5	261	166	63
6	18	5	0	209	1
1	19	5	194	164	77
2	19	5	203	137	71
3	19	5	0	65	1
4	19	5	122	117	121
5	19	5	63	16	63
0	20	5	140	71	140
1	20	5	181	151	81
2	20	5	207	168	85
0	0	6	5993	5556	15
1	0	6	1087	1307	7
2	0	6	130	214	33
3	0	6	605	570	8
4	0	6	516	450	10
5	0	6	133	47	47
6	0	6	220	215	30
7	0	6	426	428	17
8	0	6	571	556	14
9	0	6	750	733	14
10	0	6	353	285	30
11	0	6	259	167	53
12	0	6	317	299	46
13	0	6	315	302	52
14	0	6	155	104	155
1	1	6	1787	1908	4
2	1	6	1307	1351	4
3	1	6	586	821	6
4	1	6	345	288	10
5	1	6	714	655	6
6	1	6	108	121	58
7	1	6	236	306	22
8	1	6	69	77	69
9	1	6	118	118	78
10	1	6	0	21	1
11	1	6	374	319	23
12	1	6	231	150	42
13	1	6	123	33	123
14	1	6	94	74	93
0	2	6	2165	2265	6
1	2	6	1340	1406	4
2	2	6	320	285	10
3	2	6	166	172	21
4	2	6	271	297	12
5	2	6	1716	1722	5
6	2	6	186	158	24
7	2	6	275	289	18
8	2	6	278	260	21
9	2	6	0	25	1
10	2	6	66	158	66
11	2	6	0	27	1
12	2	6	222	266	42
13	2	6	393	368	28
14	2	6	400	350	30
1	3	6	729	714	7
2	3	6	667	672	5
3	3	6	1158	1199	4
4	3	6	164	178	25
5	3	6	0	44	1
6	3	6	0	17	1
7	3	6	267	204	19
8	3	6	20	31	20
9	3	6	166	134	46
10	3	6	206	184	41
11	3	6	252	272	33
12	3	6	513	482	16
13	3	6	439	427	25
14	3	6	139	232	138
0	4	6	172	229	34
1	4	6	662	629	7
2	4	6	78	115	77
3	4	6	123	151	36
4	4	6	428	432	10
5	4	6	431	384	11
6	4	6	320	305	16
7	4	6	567	497	11
8	4	6	416	381	15
9	4	6	0	114	1
10	4	6	179	110	46
11	4	6	161	33	53
12	4	6	505	473	21
13	4	6	328	314	30
14	4	6	0	18	1
1	5	6	0	142	1
2	5	6	551	529	9
3	5	6	190	266	23
4	5	6	335	299	14
5	5	6	502	466	10
6	5	6	622	634	9
7	5	6	780	749	9
8	5	6	60	44	60
9	5	6	173	207	48
10	5	6	106	96	105
11	5	6	0	4	1
12	5	6	100	115	100
13	5	6	292	316	35
14	5	6	233	278	58
0	6	6	358	215	18
1	6	6	173	84	30
2	6	6	213	243	23
3	6	6	307	365	16
4	6	6	94	67	93
5	6	6	434	387	12
6	6	6	188	234	32
7	6	6	113	134	93
8	6	6	197	151	39
9	6	6	534	489	14
10	6	6	160	138	64
11	6	6	118	28	118
12	6	6	159	154	74
13	6	6	123	102	123
14	6	6	186	245	74
1	7	6	250	172	22
2	7	6	0	50	1
3	7	6	0	107	1
4	7	6	406	400	13
5	7	6	85	82	84
6	7	6	559	551	11
7	7	6	0	11	1
8	7	6	434	437	17
9	7	6	335	273	24
10	7	6	377	441	24
11	7	6	253	260	38
12	7	6	0	14	1
13	7	6	215	147	52
0	8	6	687	620	12
1	8	6	331	344	21
2	8	6	396	354	13
3	8	6	117	84	67
4	8	6	1006	1035	6
5	8	6	870	911	8
6	8	6	78	109	77
7	8	6	533	516	15
8	8	6	0	97	1
9	8	6	0	16	1
10	8	6	57	28	56
11	8	6	131	166	131
12	8	6	252	190	42
13	8	6	122	70	121
1	9	6	248	221	25
2	9	6	112	59	71
3	9	6	768	742	8
4	9	6	670	662	9
5	9	6	453	453	15
6	9	6	606	677	11
7	9	6	192	170	39
8	9	6	72	40	72
9	9	6	151	49	68
10	9	6	0	69	1
11	9	6	100	191	100
12	9	6	125	164	125
13	9	6	92	8	91
0	10	6	0	48	1
1	10	6	0	51	1
2	10	6	35	25	34
3	10	6	175	117	38
4	10	6	614	537	12
5	10	6	367	391	19
6	10	6	304	313	23
7	10	6	108	24	107
8	10	6	244	200	38
9	10	6	159	146	71
10	10	6	155	179	80
11	10	6	0	124	1
12	10	6	108	105	107
1	11	6	60	11	60
2	11	6	393	347	19
3	11	6	245	208	29
4	11	6	147	123	57
5	11	6	503	470	15
6	11	6	200	217	40
7	11	6	0	172	1
8	11	6	483	486	19
9	11	6	238	179	42
10	11	6	0	37	1
11	11	6	92	85	91
12	11	6	147	29	147
0	12	6	128	5	128
1	12	6	78	17	77
2	12	6	260	265	32
3	12	6	189	167	47
4	12	6	0	109	1
5	12	6	497	475	16
6	12	6	198	73	41
7	12	6	384	356	24
8	12	6	352	304	28
9	12	6	353	312	30
10	12	6	128	170	128
11	12	6	108	81	107
1	13	6	0	31	1
2	13	6	268	275	35
3	13	6	206	135	52
4	13	6	227	168	38
5	13	6	146	187	68
6	13	6	521	524	18
7	13	6	272	35	37
8	13	6	260	210	41
9	13	6	0	105	1
10	13	6	269	226	42
11	13	6	0	51	1
0	14	6	0	25	1
1	14	6	446	408	19
2	14	6	127	13	126
3	14	6	134	31	134
4	14	6	281	353	34
5	14	6	638	677	16
6	14	6	163	42	70
7	14	6	78	103	77
8	14	6	0	53	1
9	14	6	399	316	27
10	14	6	0	72	1
1	15	6	232	233	47
2	15	6	142	83	141
3	15	6	200	233	61
4	15	6	326	324	36
5	15	6	418	377	27
6	15	6	0	114	1
7	15	6	234	208	46
8	15	6	0	167	1
9	15	6	0	85	1
0	16	6	180	206	127
1	16	6	268	268	41
2	16	6	0	125	1
3	16	6	179	17	81
4	16	6	274	306	48
5	16	6	0	12	1
6	16	6	94	137	93
7	16	6	271	287	42
8	16	6	0	63	1
1	17	6	239	211	50
2	17	6	186	156	72
3	17	6	85	219	84
4	17	6	104	210	104
5	17	6	0	32	1
6	17	6	0	148	1
7	17	6	0	25	1
0	18	6	348	279	48
1	18	6	125	65	125
2	18	6	104	229	104
3	18	6	66	72	66
4	18	6	0	67	1
5	18	6	0	201	1
6	18	6	130	14	129
1	19	6	158	125	117
2	19	6	0	98	1
3	19	6	142	51	141
4	19	6	280	248	59
1	1	7	820	873	5
2	1	7	971	994	4
3	1	7	523	516	7
4	1	7	118	76	35
5	1	7	551	520	8
6	1	7	104	39	75
7	1	7	75	10	74
8	1	7	331	370	18
9	1	7	0	73	1
10	1	7	0	72	1
11	1	7	0	56	1
12	1	7	290	262	33
13	1	7	0	63	1
14	1	7	192	4	82
0	2	7	723	784	6
1	2	7	0	16	1
2	2	7	830	1035	5
3	2	7	418	393	9
4	2	7	892	949	5
5	2	7	303	260	13
6	2	7	235	226	21
7	2	7	410	397	13
8	2	7	335	341	17
9	2	7	0	3	1
10	2	7	149	17	65
11	2	7	179	80	52
12	2	7	0	5	1
13	2	7	0	72	1
14	2	7	195	124	71
1	3	7	380	396	8
2	3	7	863	929	5
3	3	7	322	350	12
4	3	7	270	354	15
5	3	7	616	638	8
6	3	7	1350	1332	5
7	3	7	217	236	26
8	3	7	102	81	102
9	3	7	66	33	66
10	3	7	258	171	31
11	3	7	0	109	1
12	3	7	249	229	38
13	3	7	199	178	63
14	3	7	180	32	80
0	4	7	1304	1380	6
1	4	7	346	337	13
2	4	7	534	696	9
3	4	7	206	179	21
4	4	7	714	710	7
5	4	7	711	681	7
6	4	7	131	67	44
7	4	7	444	399	12
8	4	7	211	249	34
9	4	7	0	7	1
10	4	7	113	2	113
11	4	7	254	239	36
12	4	7	441	465	20
13	4	7	0	11	1
14	4	7	158	20	104
1	5	7	200	197	22
2	5	7	363	333	13
3	5	7	571	519	9
4	5	7	216	246	22
5	5	7	579	537	10
6	5	7	142	144	45
7	5	7	404	362	16
8	5	7	69	174	69
9	5	7	217	216	37
10	5	7	502	581	15
11	5	7	218	213	37
12	5	7	159	5	86
13	5	7	228	117	49
14	5	7	140	94	140
0	6	7	360	265	24
1	6	7	378	408	13
2	6	7	493	492	11
3	6	7	102	180	67
4	6	7	104	109	84
5	6	7	130	133	49
6	6	7	137	157	50
7	6	7	150	183	53
8	6	7	0	26	1
9	6	7	195	103	44
10	6	7	247	221	31
11	6	7	90	155	89
12	6	7	240	173	41
13	6	7	0	22	1
1	7	7	446	428	14
2	7	7	140	8	50
3	7	7	144	85	42
4	7	7	234	271	24
5	7	7	232	211	26
6	7	7	478	478	15
7	7	7	467	433	15
8	7	7	234	188	35
9	7	7	0	34	1
10	7	7	158	209	65
11	7	7	249	264	38
12	7	7	163	67	77
13	7	7	144	94	109
0	8	7	751	659	24
1	8	7	725	691	14
2	8	7	460	531	12
3	8	7	660	650	11
4	8	7	1044	1032	7
5	8	7	20	80	20
6	8	7	811	806	10
7	8	7	0	108	1
8	8	7	400	373	19
9	8	7	127	216	126
10	8	7	0	54	1
11	8	7	0	28	1
12	8	7	142	45	101
13	8	7	0	82	1
1	9	7	134	46	62
2	9	7	221	196	31
3	9	7	345	344	19
4	9	7	584	612	12
5	9	7	947	1005	7
6	9	7	100	104	100
7	9	7	149	196	65
8	9	7	0	65	1
9	9	7	0	114	1
10	9	7	289	228	34
11	9	7	229	155	46
12	9	7	143	116	108
0	10	7	180	179	70
1	10	7	233	238	31
2	10	7	511	525	13
3	10	7	218	213	30
4	10	7	606	610	13
5	10	7	57	74	56
6	10	7	53	76	52
7	10	7	0	11	1
8	10	7	271	213	32
9	10	7	257	246	36
10	10	7	173	143	67
11	10	7	0	17	1
12	10	7	225	191	51
1	11	7	0	17	1
2	11	7	350	312	21
3	11	7	345	334	20
4	11	7	35	87	34
5	11	7	165	255	55
6	11	7	224	217	36
7	11	7	163	164	58
8	11	7	75	238	74
9	11	7	329	356	29
10	11	7	0	3	1
11	11	7	166	169	83
12	11	7	0	32	1
0	12	7	247	201	46
1	12	7	213	151	39
2	12	7	0	79	1
3	12	7	0	50	1
4	12	7	238	151	37
5	12	7	269	224	30
6	12	7	178	291	51
7	12	7	0	21	1
8	12	7	181	117	61
9	12	7	482	487	19
10	12	7	72	79	72
11	12	7	137	50	137
1	13	7	228	189	38
2	13	7	94	65	93

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
3	13	7	57	7	56
4	13	7	288	256	30
5	13	7	411	497	21
6	13	7	332	304	31
7	13	7	213	195	47
8	13	7	315	348	32
9	13	7	123	101	123
10	13	7	161	19	98
0	14	7	0	157	1
1	14	7	40	82	40
2	14	7	0	148	1
3	14	7	384	359	27
4	14	7	262	229	38
5	14	7	0	125	1
6	14	7	247	236	38
7	14	7	137	256	108
8	14	7	343	344	31
9	14	7	0	2	1
10	14	7	303	329	41
1	15	7	122	56	121
2	15	7	0	104	1
3	15	7	146	94	145
4	15	7	539	457	22
5	15	7	227	159	48
6	15	7	72	68	72
7	15	7	0	57	1
8	15	7	180	194	70
9	15	7	0	98	1
0	16	7	0	168	1
1	16	7	100	171	100
2	16	7	230	114	50
3	16	7	113	42	113
4	16	7	106	99	105
5	16	7	0	90	1
6	16	7	63	43	63
7	16	7	171	99	83
8	16	7	0	120	1
1	17	7	182	113	74
2	17	7	0	28	1
3	17	7	292	358	42
4	17	7	265	227	51
5	17	7	246	276	69
6	17	7	214	292	63
7	17	7	98	69	98
0	18	7	172	137	172
1	18	7	173	175	88
2	18	7	0	95	1
3	18	7	0	99	1
4	18	7	96	23	96
5	18	7	0	62	1
1	19	7	161	184	141
2	19	7	117	87	116
3	19	7	234	90	58
0	0	8	535	446	11
1	0	8	685	626	8
2	0	8	139	112	34
3	0	8	1605	1806	6
4	0	8	563	574	10
5	0	8	1035	1058	8
6	0	8	527	465	15
7	0	8	456	476	17
8	0	8	280	315	31
9	0	8	20	24	20
10	0	8	503	478	22
11	0	8	419	360	32
12	0	8	87	15	87
13	0	8	0	68	1
14	0	8	321	238	53
1	1	8	441	376	7
2	1	8	225	256	16
3	1	8	1134	1250	5
4	1	8	807	815	5
5	1	8	471	428	12
6	1	8	75	28	74
7	1	8	468	454	13
8	1	8	306	340	21
9	1	8	159	118	49
10	1	8	432	389	19
11	1	8	28	51	28
12	1	8	315	235	32
13	1	8	194	190	62
14	1	8	0	58	1
0	2	8	231	200	17
1	2	8	249	244	15
2	2	8	120	67	35
3	2	8	60	59	60
4	2	8	255	280	16
5	2	8	194	215	24
6	2	8	449	381	11
7	2	8	113	107	64
8	2	8	298	292	21
9	2	8	302	335	24
10	2	8	195	213	45
11	2	8	28	33	28
12	2	8	205	222	54
13	2	8	53	149	52
14	2	8	104	127	104
1	3	8	211	230	15
2	3	8	310	340	13
3	3	8	394	397	9
4	3	8	0	61	1
5	3	8	341	303	13
6	3	8	240	226	22
7	3	8	108	124	79
8	3	8	102	73	102
9	3	8	200	218	40
10	3	8	0	39	1
11	3	8	0	11	1
12	3	8	240	222	39
13	3	8	321	369	37
14	3	8	137	74	137
0	4	8	202	198	34
1	4	8	223	195	18
2	4	8	653	671	7
3	4	8	0	64	1
4	4	8	219	222	21
5	4	8	80	19	80
6	4	8	144	26	46
7	4	8	218	222	27
8	4	8	398	353	19
9	4	8	98	45	98
10	4	8	341	330	24
11	4	8	258	215	35
12	4	8	269	303	35
13	4	8	151	8	111
1	5	8	487	506	10
2	5	8	28	135	28
3	5	8	539	611	10
4	5	8	870	822	7
5	5	8	954	908	6
6	5	8	146	124	45
7	5	8	190	212	36
8	5	8	0	7	1
9	5	8	503	422	16
10	5	8	123	194	123
11	5	8	130	191	129
12	5	8	503	508	18
13	5	8	0	78	1
0	6	8	408	406	22
1	6	8	446	447	12
2	6	8	808	852	8
3	6	8	796	807	7
4	6	8	87	32	87
5	6	8	592	589	11
6	6	8	400	381	14
7	6	8	287	340	24
8	6	8	66	51	66
9	6	8	159	253	60
10	6	8	450	473	20
11	6	8	438	400	24
12	6	8	78	26	77
13	6	8	137	124	137
1	7	8	513	551	12
2	7	8	92	157	91
3	7	8	280	284	21
4	7	8	234	258	27
5	7	8	416	381	16
6	7	8	342	355	19
7	7	8	706	670	13
8	7	8	369	341	21
9	7	8	475	369	19
10	7	8	288	266	28
11	7	8	258	289	41
12	7	8	154	187	87
13	7	8	185	146	64
0	8	8	533	465	18
1	8	8	96	59	96
2	8	8	439	406	15
3	8	8	0	57	1
4	8	8	616	648	10
5	8	8	322	358	19
6	8	8	960	955	9
7	8	8	96	89	96
8	8	8	248	172	32
9	8	8	533	488	16
10	8	8	240	258	42
11	8	8	0	57	1
12	8	8	252	180	43
1	9	8	0	53	1
2	9	8	40	106	40
3	9	8	0	47	1
4	9	8	108	49	107
5	9	8	655	636	12
6	9	8	356	323	21
7	9	8	49	125	49
8	9	8	0	24	1
9	9	8	0	23	1
10	9	8	53	71	52
11	9	8	115	41	115
12	9	8	102	78	102
0	10	8	63	94	63
1	10	8	203	223	39
2	10	8	160	16	56
3	10	8	108	181	107
4	10	8	234	243	33
5	10	8	131	181	64
6	10	8	393	389	18
7	10	8	130	202	101
8	10	8	223	209	40
9	10	8	189	226	57
10	10	8	210	195	52
11	10	8	169	69	76
12	10	8	222	26	49
1	11	8	127	139	86
2	11	8	294	273	27
3	11	8	155	154	67
4	11	8	0	52	1
5	11	8	60	142	60
6	11	8	0	32	1
7	11	8	172	6	58
8	11	8	373	407	23
9	11	8	525	570	19
10	11	8	191	253	61
11	11	8	104	87	104
0	12	8	221	34	51
1	12	8	208	164	40
2	12	8	102	99	102
3	12	8	161	118	61
4	12	8	57	3	56
5	12	8	63	17	63
6	12	8	317	299	27
7	12	8	323	342	28
8	12	8	531	522	18
9	12	8	319	374	32
10	12	8	198	125	60
11	12	8	28	88	28
1	13	8	144	44	75
2	13	8	176	160	62
3	13	8	403	358	24
4	13	8	0	25	1
5	13	8	0	9	1
6	13	8	158	139	70
7	13	8	300	233	33
8	13	8	194	230	55
9	13	8	140	163	140
10	13	8	87	242	87
0	14	8	228	354	69
1	14	8	155	40	82
2	14	8	320	310	30
3	14	8	158	186	85
4	14	8	232	93	43
5	14	8	66	47	66
6	14	8	302	338	30
7	14	8	238	299	40
8	14	8	229	160	48
9	14	8	389	385	30
1	15	8	0	12	1
2	15	8	127	150	126
3	15	8	395	357	28
4	15	8	207	145	66
5	15	8	0	40	1
6	15	8	235	313	50
7	15	8	127	3	126
8	15	8	239	250	47
0	16	8	362	320	46
1	16	8	63	63	63
2	16	8	334	319	34
3	16	8	0	14	1
4	16	8	227	275	61
5	16	8	213	209	68
6	16	8	0	84	1
7	16	8	0	94	1
1	17	8	255	270	48
2	17	8	154	49	109
3	17	8	123	34	123
4	17	8	300	332	45
5	17	8	150	61	149
6	17	8	0	71	1
0	18	8	172	84	172
1	18	8	0	96	1
2	18	8	90	1	89
3	18	8	266	259	47
4	18	8	203	169	91
5	18	8	173	239	173
1	19	8	173	2	113
2	19	8	140	56	140
1	1	9	487	487	7
2	1	9	825	841	5
3	1	9	438	459	9
4	1	9	217	221	20
5	1	9	313	284	15
6	1	9	390	366	14
7	1	9	220	157	31
8	1	9	332	351	21
9	1	9	194	213	41
10	1	9	275	265	31
11	1	9	159	181	78
12	1	9	115	51	115
13	1	9	0	64	1
0	2	9	1382	1469	8
1	2	9	642	590	6
2	2	9	618	670	9
3	2	9	448	459	9
4	2	9	85	69	84
5	2	9	305	286	15
6	2	9	302	260	18
7	2	9	564	523	12
8	2	9	352	279	19
9	2	9	617	559	12
10	2	9	53	48	52
11	2	9	233	321	43
12	2	9	229	268	47
13	2	9	0	45	1
1	3	9	732	679	6
2	3	9	0	5	1
3	3	9	113	69	43
4	3	9	294	292	16
5	3	9	404	377	12
6	3	9	172	146	36
7	3	9	210	199	32
8	3	9	173	206	46
9	3	9	147	85	67
10	3	9	146	155	82
11	3	9	300	335	30
12	3	9	182	125	67
13	3	9	165	5	95
0	4	9	836	880	8
1	4	9	244	259	17
2	4	9	115	171	45
3	4	9	40	92	40
4	4	9	315	297	16
5	4	9	207	204	25
6	4	9	292	288	19
7	4	9	204	111	33
8	4	9	137	19	67
9	4	9	329	286	26
10	4	9	115	93	115
11	4	9	144	93	84
12	4	9	252	241	40
13	4	9	0	79	1
1	5	9	0	38	1
2	5	9	302	337	19
3	5	9	765	749	8
4	5	9	558	547	10
5	5	9	57	91	56
6	5	9	113	144	78
7	5	9	208	272	33
8	5	9	218	242	38
9	5	9	514	482	17
10	5	9	40	41	40
11	5	9	505	479	20
12	5	9	245	264	46
13	5	9	329	289	33
0	6	9	187	179	50
1	6	9	349	317	15
2	6	9	186	243	33
3	6	9	195	175	30
4	6	9	35	160	34
5	6	9	447	454	
6	6	9	330	333	
7	6	9	0	20	
8	6	9	134	117	8
9	6	9	460	453	2
10	6	9	457	457	20
11	6	9	280	301	33
12	6	9	155	84	77
13	6	9	290	196	44
1	7	9	385	359	18
2	7	9	258	238	22
3	7	9	0	75	1
4	7	9	343	297	18
5	7	9	401	412	16
6	7	9	282	233	25
7	7	9	247	230	30
8	7	9	69	131	69
9	7	9	171	114	57
10	7	9	149	190	82
11	7	9	151	142	78
12	7	9	130	202	129
0	8	9	83	31	82
1	8	9	156	97	52
2	8	9	453	452	14
3	8	9	411	414	16
4	8	9	486	497	13
5	8	9	125	166	80
6	8	9	341	402	23
7	8	9	196	257	43
8	8	9	424	406	20
9	8	9	568	532	18
10	8	9	388	355	21
11	8	9	0	68	1
12	8	9	249	132	46
1	9	9	574	557	12
2	9	9	667	608	11
3	9	9	0	34	1
4	9	9	0	22	1
5	9	9	0	77	1
6	9	9	0	6	1
7	9	9	323	312	25
8	9	9	0	135	1
9	9	9	154	6	71
10	9	9	115	87	115
11	9	9	216	206	53
12	9	9	72	130	72
0	10	9	245	161	49
1	10	9	241	292	32
2	10	9	279	244	30
3	10	9	230	196	34
4	10	9	149	171	65
5	10	9	0	106	1
6	10	9	465	527	17
7	10	9	298	364	28
8	10	9	397	520	21
9	10	9	0	77	1
10	10	9	0	126	1
11	10	9	289	299	42
1	11	9	80	80	80
2	11	9	189	195	45
3	11	9	328	322	26
4	11	9	0	66	1
5	11	9	0	16	1
6	11	9	239	226	37
7	11	9	443	530	22
8	11	9	234	219	42
9	11	9	410	345	25
10	11	9	346	385	32
11	11	9	20	37	20
0	12	9	85	119	84
1	12	9	0	24	1
2	12	9	172	29	57
3	12	9	0	69	1
4	12	9	0	15	1
5	12	9	106	11	105
6	12	9	125	96	125
7	12	9	0	104	1
8	12	9	348	321	28
9	12	9	218	176	52
10	12	9	244	204	50
1	13	9	0	54	1
2	13	9	0	79	1
3	13	9	227	223	46
4	13	9	245	196	39
5	13	9	0	56	1
6	13	9	290	238	33
7	13	9	412	350	24

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
8	13	9	130	149	129
9	13	9	496	463	26
10	13	9	191	274	70
0	14	9	175	60	108
1	14	9	0	16	1
2	14	9	156	182	84
3	14	9	170	3	80
4	14	9	182	159	63
5	14	9	0	28	1
6	14	9	0	51	1
7	14	9	110	137	109
8	14	9	191	203	69
9	14	9	0	129	1
1	15	9	330	379	36
2	15	9	0	15	1
3	15	9	488	444	23
4	15	9	0	13	1
5	15	9	294	278	34
6	15	9	100	12	100
7	15	9	0	7	1
8	15	9	0	97	1
0	16	9	424	371	37
1	16	9	0	93	1
2	16	9	443	424	25
3	16	9	199	17	67
4	16	9	213	203	71
5	16	9	0	76	1
6	16	9	0	52	1
7	16	9	112	179	111
1	17	9	0	183	1
2	17	9	0	34	1
3	17	9	611	552	20
4	17	9	204	147	77
5	17	9	75	64	74
6	17	9	139	161	138
0	18	9	172	20	172
1	18	9	0	11	1
2	18	9	143	93	143
3	18	9	85	152	84
4	18	9	0	130	1
0	0	10	1190	1110	11
1	0	10	288	284	22
2	0	10	507	548	14
3	0	10	94	141	93
4	0	10	536	481	15
5	0	10	491	464	16
6	0	10	130	16	109
7	0	10	85	15	84
8	0	10	583	520	18
9	0	10	322	280	35
10	0	10	165	44	165
11	0	10	297	93	51
12	0	10	337	203	52
13	0	10	78	274	77
1	1	10	275	303	14
2	1	10	1164	1149	5
3	1	10	193	201	23
4	1	10	254	264	17
5	1	10	420	401	12
6	1	10	112	122	83
7	1	10	404	353	17
8	1	10	0	1	1
9	1	10	279	295	28
10	1	10	104	137	104
11	1	10	0	86	1
12	1	10	272	113	45
13	1	10	0	13	1
0	2	10	0	35	1
1	2	10	146	164	32
2	2	10	388	361	11
3	2	10	28	51	28
4	2	10	297	301	15
5	2	10	0	90	1
6	2	10	60	69	60
7	2	10	232	219	29
8	2	10	166	147	49
9	2	10	591	607	14
10	2	10	0	13	1
11	2	10	90	12	89
12	2	10	386	363	29
13	2	10	176	112	86
1	3	10	227	227	21
2	3	10	296	274	16
3	3	10	310	307	16
4	3	10	154	101	33
5	3	10	0	88	1
6	3	10	189	203	35
7	3	10	351	294	19
8	3	10	106	96	105
9	3	10	127	57	126
10	3	10	102	17	102
11	3	10	0	86	1
12	3	10	228	264	52
13	3	10	100	40	100
0	4	10	237	288	38
1	4	10	671	604	6
2	4	10	102	155	53
3	4	10	0	76	1
4	4	10	429	373	12
5	4	10	0	8	1
6	4	10	290	213	22
7	4	10	0	21	1
8	4	10	204	129	42
9	4	10	365	306	25
10	4	10	98	75	98
11	4	10	0	64	1
12	4	10	221	253	53
13	4	10	176	206	95
1	5	10	462	369	11
2	5	10	243	229	18
3	5	10	301	301	18
4	5	10	421	418	12
5	5	10	483	420	11
6	5	10	400	389	16
7	5	10	296	312	24
8	5	10	0	39	1
9	5	10	330	262	26
10	5	10	381	354	25
11	5	10	337	241	31
12	5	10	230	142	48
13	5	10	155	140	155
0	6	10	182	209	55
1	6	10	49	23	49
2	6	10	657	673	8
3	6	10	0	11	1
4	6	10	352	332	15
5	6	10	189	200	36
6	6	10	106	115	105
7	6	10	279	297	28
8	6	10	447	379	20
9	6	10	0	6	1
10	6	10	0	53	1
11	6	10	232	183	46
12	6	10	0	44	1
1	7	10	340	350	20
2	7	10	297	316	19
3	7	10	361	348	18
4	7	10	545	528	13
5	7	10	164	150	47
6	7	10	676	666	12
7	7	10	508	444	18
8	7	10	140	176	83
9	7	10	108	203	107
10	7	10	496	398	19
11	7	10	120	158	120
12	7	10	151	30	87
0	8	10	584	592	18
1	8	10	200	155	38
2	8	10	427	411	15
3	8	10	53	52	52
4	8	10	326	295	24
5	8	10	460	429	16
6	8	10	0	60	1
7	8	10	117	118	116
8	8	10	136	14	101
9	8	10	445	471	20
10	8	10	0	26	1
11	8	10	0	46	1
12	8	10	128	123	128
1	9	10	154	37	62
2	9	10	0	37	1
3	9	10	401	361	18
4	9	10	268	215	28
5	9	10	0	153	1
6	9	10	364	309	24
7	9	10	439	489	21
8	9	10	200	136	54
9	9	10	325	347	29
10	9	10	0	52	1
11	9	10	239	8	46
0	10	10	0	142	1
1	10	10	161	168	53
2	10	10	80	138	80
3	10	10	133	172	83
4	10	10	242	221	37
5	10	10	0	24	1
6	10	10	204	193	46
7	10	10	302	277	32
8	10	10	0	74	1
9	10	10	182	93	60
10	10	10	186	91	76
11	10	10	312	303	35
1	11	10	0	37	1
2	11	10	229	232	38
3	11	10	102	163	102
4	11	10	196	200	50
5	11	10	199	180	48
6	11	10	122	116	121
7	11	10	131	230	131
8	11	10	520	551	22
9	11	10	218	226	54
10	11	10	266	248	41
0	12	10	0	66	1
1	12	10	0	39	1
2	12	10	440	369	19
3	12	10	110	158	109
4	12	10	150	121	83
5	12	10	120	133	120
6	12	10	275	275	36
7	12	10	272	344	35
8	12	10	234	193	44
9	12	10	0	3	1
10	12	10	102	48	102
1	13	10	317	297	28
2	13	10	0	53	1
3	13	10	35	33	34
4	13	10	199	199	54
5	13	10	75	153	74
6	13	10	0	26	1
7	13	10	134	128	134
8	13	10	356	401	29
9	13	10	322	345	39
0	14	10	260	234	62
1	14	10	75	22	74
2	14	10	253	212	38
3	14	10	100	218	100
4	14	10	298	250	37
5	14	10	49	63	49
6	14	10	131	89	131
7	14	10	0	51	1
8	14	10	262	239	45
1	15	10	186	89	67
2	15	10	195	122	62
3	15	10	169	20	81
4	15	10	229	80	60
5	15	10	245	302	45
6	15	10	0	96	1
7	15	10	0	80	1
0	16	10	226	281	78
1	16	10	151	95	111
2	16	10	117	184	116
3	16	10	219	278	57
4	16	10	0	95	1
5	16	10	118	67	118
6	16	10	0	39	1
1	17	10	298	298	40
2	17	10	0	45	1
3	17	10	0	15	1
4	17	10	0	101	1
5	17	10	212	126	77
0	18	10	365	152	48
1	18	10	0	112	1
2	18	10	272	287	51
1	1	11	0	31	1
2	1	11	299	281	14
3	1	11	90	140	89
4	1	11	563	508	9
5	1	11	424	414	13
6	1	11	248	278	27
7	1	11	181	216	41
8	1	11	290	275	28
9	1	11	0	113	1
10	1	11	432	341	28
11	1	11	187	238	82
12	1	11	0	172	1
13	1	11	196	43	84
0	2	11	471	456	22
1	2	11	599	625	7
2	2	11	630	654	8
3	2	11	184	224	28
4	2	11	379	349	14
5	2	11	0	74	1
6	2	11	60	80	60
7	2	11	333	278	24
8	2	11	140	33	83
9	2	11	85	13	84
10	2	11	475	415	23
11	2	11	0	174	1
12	2	11	161	81	121
13	2	11	0	80	1
1	3	11	455	454	11
2	3	11	78	114	77
3	3	11	461	454	11
4	3	11	66	118	66
5	3	11	243	269	25
6	3	11	338	301	19
7	3	11	66	122	66
8	3	11	0	40	1
9	3	11	0	34	1
10	3	11	0	94	1
11	3	11	0	42	1
12	3	11	63	3	63
0	4	11	502	505	13
1	4	11	532	544	10
2	4	11	612	599	9
3	4	11	92	121	91
4	4	11	203	162	28
5	4	11	176	121	36
6	4	11	350	290	21
7	4	11	212	185	42
8	4	11	247	202	37
9	4	11	272	222	33
10	4	11	194	224	58
11	4	11	134	5	134
12	4	11	214	242	65
1	5	11	0	72	1
2	5	11	192	145	28
3	5	11	227	251	25
4	5	11	66	59	66
5	5	11	0	67	1
6	5	11	283	238	25
7	5	11	0	3	1
8	5	11	297	296	29
9	5	11	108	195	107
10	5	11	304	275	33
11	5	11	430	413	23
12	5	11	110	238	109
0	6	11	0	27	1
1	6	11	234	198	25
2	6	11	187	152	30
3	6	11	0	76	1
4	6	11	364	402	18
5	6	11	140	34	57
6	6	11	96	36	96
7	6	11	321	333	25
8	6	11	0	91	1
9	6	11	193	198	54
10	6	11	115	53	115
11	6	11	49	171	49
12	6	11	0	9	1
1	7	11	0	16	1
2	7	11	373	377	17
3	7	11	241	253	28
4	7	11	436	435	15
5	7	11	164	222	52
6	7	11	100	202	100
7	7	11	237	177	37
8	7	11	87	37	87
9	7	11	423	453	22
10	7	11	0	177	1
11	7	11	160	153	80
12	7	11	253	217	51
0	8	11	199	162	66
1	8	11	0	32	1
2	8	11	250	230	27
3	8	11	153	134	56
4	8	11	0	115	1
5	8	11	184	133	52
6	8	11	329	361	25
7	8	11	94	59	93
8	8	11	305	338	28
9	8	11	234	244	43
10	8	11	489	481	20
11	8	11	231	167	51
1	9	11	83	22	82
2	9	11	0	65	1
3	9	11	365	319	23
4	9	11	326	399	26
5	9	11	166	2	62
6	9	11	131	182	131
7	9	11	143	144	86
8	9	11	629	668	17
9	9	11	57	16	56
10	9	11	0	70	1
11	9	11	146	177	117
0	10	11	318	288	41
1	10	11	120	161	120
2	10	11	139	94	75
3	10	11	69	63	69
4	10	11	140	64	91
5	10	11	177	144	63
6	10	11	338	306	33
7	10	11	0	54	1
8	10	11	0	112	1
9	10	11	192	6	59
10	10	11	0	99	1
1	11	11	161	34	65
2	11	11	96	137	96
3	11	11	214	232	42
4	11	11	226	111	43
5	11	11	28	62	28
6	11	11	0	19	1
7	11	11	665	621	16
8	11	11	732	729	14
9	11	11	125	38	125
10	11	11	322	304	34
0	12	11	0	76	1
1	12	11	28	97	28
2	12	11	0	27	1
3	12	11	0	81	1
4	12	11	136	94	135
5	12	11	0	55	1
6	12	11	435	405	22
7	12	11	453	493	21
8	12	11	522	540	20
9	12	11	181	277	75
1	13	11	0	78	1
2	13	11	0	7	1
3	13	11	0	107	1
4	13	11	237	203	45
5	13	11	275	217	38
6	13	11	0	13	1
7	13	11	338	320	29
8	13	11	418	365	26
9	13	11	203	204	67
0	14	11	0	126	1
1	14	11	106	120	105
2	14	11	178	107	70
3	14	11	92	170	91
4	14	11	210	182	62
5	14	11	85	114	84
6	14	11	0	42	1
7	14	11	0	56	1
8	14	11	232	256	50
1	15	11	0	16	1
2	15	11	155	159	102
3	15	11	153	4	124
4	15	11	0	91	1
5	15	11	120	90	120
6	15	11	0	79	1
7	15	11	102	10	102
0	16	11	0	119	1
1	16	11	200	208	67
2	16	11	0	135	1
3	16	11	83	73	82
4	16	11	130	45	129
5	16	11	159	187	124
1	17	11	143	270	143
2	17	11	206	87	73
3	17	11	0	69	1
4	17	11	137	177	137
0	0	12	437	442	33
1	0	12	1007	995	9
2	0	12	0	15	1
3	0	12	1024	973	9
4	0	12	120	131	120
5	0	12	371	381	27
6	0	12	567	475	21
7	0	12	255	245	52
8	0	12	234	92	56
9	0	12	20	109	20
10	0	12	150	165	149
11	0	12	147	105	147
12	0	12	300	302	69
1	1	12	1259	1296	5

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
2	1	12	697	704	9
3	1	12	387	370	16
4	1	12	831	803	8
5	1	12	569	508	13
6	1	12	369	361	21
7	1	12	185	183	53
8	1	12	0	16	1
9	1	12	156	113	93
10	1	12	219	97	62
11	1	12	215	182	67
12	1	12	149	17	148
0	2	12	361	275	30
1	2	12	549	531	9
2	2	12	484	472	11
3	2	12	92	41	91
4	2	12	171	130	41
5	2	12	186	175	39
6	2	12	130	192	101
7	2	12	186	90	54
8	2	12	35	91	34
9	2	12	257	218	45
10	2	12	485	515	22
11	2	12	0	172	1
12	2	12	80	48	80
1	3	12	168	188	34
2	3	12	72	60	72
3	3	12	96	71	96
4	3	12	338	345	17
5	3	12	35	73	34
6	3	12	158	175	59
7	3	12	0	41	1
8	3	12	227	193	50
9	3	12	117	38	116
10	3	12	0	75	1
11	3	12	260	262	47
12	3	12	159	193	158
0	4	12	280	270	42
1	4	12	0	69	1
2	4	12	209	169	27
3	4	12	0	92	1
4	4	12	0	131	1
5	4	12	0	29	1
6	4	12	163	204	53
7	4	12	0	164	1
8	4	12	175	136	72
9	4	12	173	116	83
10	4	12	0	105	1
11	4	12	414	442	30
12	4	12	251	188	57
1	5	12	253	251	25
2	5	12	66	121	66
3	5	12	178	127	36
4	5	12	458	423	13
5	5	12	442	372	16
6	5	12	356	257	21
7	5	12	211	172	44
8	5	12	90	244	89
9	5	12	0	151	1
10	5	12	241	236	49
11	5	12	175	15	85
12	5	12	120	23	120
0	6	12	796	769	16
1	6	12	432	440	15
2	6	12	216	165	33
3	6	12	104	50	104
4	6	12	150	200	51
5	6	12	253	196	29
6	6	12	0	39	1
7	6	12	471	512	20
8	6	12	0	106	1
9	6	12	0	11	1
10	6	12	163	188	99
11	6	12	0	158	1
1	7	12	219	195	33
2	7	12	0	139	1
3	7	12	403	398	17
4	7	12	0	38	1
5	7	12	231	142	36
6	7	12	321	290	26
7	7	12	0	214	1
8	7	12	499	531	20
9	7	12	0	2	1
10	7	12	260	263	44
11	7	12	0	117	1
0	8	12	221	172	72
1	8	12	0	17	1
2	8	12	133	125	69
3	8	12	197	225	39
4	8	12	179	210	49
5	8	12	220	245	40
6	8	12	189	204	53
7	8	12	346	371	26
8	8	12	100	36	100
9	8	12	474	529	20
10	8	12	0	63	1
11	8	12	220	92	65
1	9	12	0	149	1
2	9	12	0	14	1
3	9	12	153	176	69
4	9	12	0	95	1
5	9	12	0	56	1
6	9	12	0	149	1
7	9	12	175	147	61
8	9	12	609	660	16
9	9	12	133	165	132
10	9	12	0	84	1
0	10	12	0	28	1
1	10	12	35	10	34
2	10	12	0	51	1
3	10	12	173	163	58
4	10	12	0	87	1
5	10	12	60	219	60
6	10	12	385	414	25
7	10	12	688	733	14
8	10	12	289	274	36
9	10	12	0	97	1
10	10	12	113	104	113
1	11	12	0	9	1
2	11	12	120	23	120
3	11	12	0	4	1
4	11	12	224	247	48
5	11	12	490	452	20
6	11	12	450	459	23
7	11	12	339	303	31
8	11	12	497	479	22
9	11	12	263	217	37
0	12	12	327	357	54
1	12	12	66	118	66
2	12	12	0	54	1
3	12	12	0	69	1
4	12	12	181	162	62
5	12	12	253	196	41
6	12	12	146	19	111
7	12	12	230	239	46
8	12	12	45	100	44
9	12	12	475	423	27
1	13	12	0	6	1
2	13	12	227	161	59
3	13	12	120	34	120
4	13	12	136	41	135
5	13	12	0	60	1
6	13	12	92	27	91
7	13	12	0	21	1
8	13	12	149	125	148
0	14	12	0	8	1
1	14	12	0	60	1
2	14	12	100	33	100
3	14	12	0	20	1
4	14	12	178	242	81
5	14	12	253	314	44
6	14	12	110	226	109
7	14	12	142	15	141
1	15	12	133	15	132
2	15	12	0	73	1
3	15	12	115	113	115
4	15	12	0	60	1
5	15	12	20	39	20
6	15	12	122	151	121
0	16	12	0	154	1
1	16	12	0	10	1
2	16	12	131	93	131
3	16	12	96	12	96
4	16	12	117	95	116
1	17	12	0	62	1
2	17	12	274	187	53
1	1	13	0	8	1
2	1	13	92	151	91
3	1	13	211	219	34
4	1	13	161	122	49
5	1	13	0	18	1
6	1	13	178	230	51
7	1	13	94	10	93
8	1	13	144	166	95
9	1	13	0	81	1
10	1	13	221	129	62
11	1	13	170	143	106
12	1	13	0	128	1
0	2	13	1448	1348	11
1	2	13	0	171	1
2	2	13	186	180	37
3	2	13	123	192	74
4	2	13	87	38	87
5	2	13	150	122	62
6	2	13	94	117	93
7	2	13	292	244	32
8	2	13	0	26	1
9	2	13	254	197	45
10	2	13	78	93	77
11	2	13	0	2	1
12	2	13	87	74	87
1	3	13	473	474	15
2	3	13	0	29	1
3	3	13	296	261	23
4	3	13	227	159	32
5	3	13	284	190	30
6	3	13	249	252	36
7	3	13	264	294	37
8	3	13	78	136	77
9	3	13	198	186	70
10	3	13	0	17	1
11	3	13	0	18	1
12	3	13	228	180	69
0	4	13	321	338	42
1	4	13	201	182	42
2	4	13	277	282	28
3	4	13	0	88	1
4	4	13	189	200	39
5	4	13	168	161	52
6	4	13	0	50	1
7	4	13	397	266	28
8	4	13	379	283	31
9	4	13	0	168	1
10	4	13	194	153	70
11	4	13	0	120	1
1	5	13	0	17	1
2	5	13	200	184	37
3	5	13	0	41	1
4	5	13	154	139	57
5	5	13	197	157	44
6	5	13	0	171	1
7	5	13	381	347	27
8	5	13	144	13	116
9	5	13	85	14	84
10	5	13	49	93	49
11	5	13	0	16	1
0	6	13	344	396	36
1	6	13	0	49	1
2	6	13	0	70	1
3	6	13	142	129	54
4	6	13	169	158	53
5	6	13	151	131	71
6	6	13	96	146	96
7	6	13	0	13	1
8	6	13	72	134	72
9	6	13	53	24	52
10	6	13	100	76	100
11	6	13	100	78	100
1	7	13	0	82	1
2	7	13	176	73	47
3	7	13	177	163	50
4	7	13	178	164	46
5	7	13	0	169	1
6	7	13	125	23	125
7	7	13	354	323	30
8	7	13	209	194	55
9	7	13	213	15	55
10	7	13	223	238	59
11	7	13	0	72	1
0	8	13	0	41	1
1	8	13	0	6	1
2	8	13	123	26	123
3	8	13	0	65	1
4	8	13	345	304	23
5	8	13	69	11	69
6	8	13	226	154	46
7	8	13	182	209	70
8	8	13	285	275	40
9	8	13	0	74	1
10	8	13	120	23	120
1	9	13	0	162	1
2	9	13	266	257	34
3	9	13	160	138	66
4	9	13	118	204	118
5	9	13	239	290	42
6	9	13	285	287	34
7	9	13	201	156	59
8	9	13	0	100	1
9	9	13	0	34	1
10	9	13	278	231	43
0	10	13	87	48	87
1	10	13	398	315	27
2	10	13	168	136	73
3	10	13	0	48	1
4	10	13	467	433	22
5	10	13	0	36	1
6	10	13	509	582	19
7	10	13	200	193	54
8	10	13	266	234	42
9	10	13	242	158	51
1	11	13	133	18	132
2	11	13	0	35	1
3	11	13	83	104	82
4	11	13	257	251	47
5	11	13	361	341	30
6	11	13	488	501	23
7	11	13	206	215	53
8	11	13	218	54	58
9	11	13	158	161	117
0	12	13	169	139	168
1	12	13	20	89	20
2	12	13	49	49	49
3	12	13	0	33	1
4	12	13	192	40	70
5	12	13	197	174	64
6	12	13	123	76	123
7	12	13	383	375	31
8	12	13	218	107	51
1	13	13	0	0	1
2	13	13	0	68	1
3	13	13	171	26	98
4	13	13	98	78	98
5	13	13	197	272	64
6	13	13	0	99	1
7	13	13	202	55	59
0	14	13	0	37	1
1	14	13	0	68	1
2	14	13	137	53	137
3	14	13	228	206	56
4	14	13	254	157	57
5	14	13	0	30	1
6	14	13	172	141	105
1	15	13	0	149	1
2	15	13	112	172	111
3	15	13	0	46	1
4	15	13	0	117	1
5	15	13	92	18	91
0	16	13	266	41	76
1	16	13	193	118	97
2	16	13	324	262	41
3	16	13	125	62	125
0	0	14	0	110	1
1	0	14	154	175	153
2	0	14	213	203	77
3	0	14	0	19	1
4	0	14	485	497	22
5	0	14	282	268	48
6	0	14	189	43	92
7	0	14	251	246	64
8	0	14	245	260	63
9	0	14	0	7	1
10	0	14	0	50	1
11	0	14	285	331	78
1	1	14	395	417	23
2	1	14	387	382	24
3	1	14	518	537	15
4	1	14	204	195	41
5	1	14	209	235	52
6	1	14	0	56	1
7	1	14	0	10	1
8	1	14	0	23	1
9	1	14	215	21	60
10	1	14	156	24	156
11	1	14	203	157	83
0	2	14	175	129	94
1	2	14	0	11	1
2	2	14	0	92	1
3	2	14	207	162	42
4	2	14	110	83	109
5	2	14	0	56	1
6	2	14	134	92	99
7	2	14	173	92	67
8	2	14	108	194	107
9	2	14	176	100	88
10	2	14	92	52	91
11	2	14	45	79	44
1	3	14	0	67	1
2	3	14	199	114	45
3	3	14	317	247	26
4	3	14	45	66	44
5	3	14	94	137	93
6	3	14	0	42	1
7	3	14	85	64	84
8	3	14	98	44	98
9	3	14	163	97	102
10	3	14	0	126	1
11	3	14	164	123	123
0	4	14	0	52	1
1	4	14	92	87	91
2	4	14	375	361	21
3	4	14	0	6	1
4	4	14	284	226	32
5	4	14	478	487	20
6	4	14	28	105	28
7	4	14	260	266	42
8	4	14	195	175	70
9	4	14	85	118	84
10	4	14	87	150	87
11	4	14	249	301	61
1	5	14	0	56	1
2	5	14	250	229	36
3	5	14	378	401	24
4	5	14	0	3	1
5	5	14	149	154	85
6	5	14	325	290	31
7	5	14	0	70	1
8	5	14	308	270	38
9	5	14	282	259	44
10	5	14	228	288	62
11	5	14	186	176	96
0	6	14	100	100	100
1	6	14	0	6	1
2	6	14	0	42	1
3	6	14	0	1	1
4	6	14	390	366	24
5	6	14	151	56	87
6	6	14	198	207	59
7	6	14	271	210	41
8	6	14	0	25	1
9	6	14	125	58	125
10	6	14	0	113	1
1	7	14	0	94	1
2	7	14	0	83	1
3	7	14	188	99	53
4	7	14	212	155	51
5	7	14	0	47	1
6	7	14	172	204	76
7	7	14	125	108	125
8	7	14	130	159	129
9	7	14	154	149	119
10	7	14	0	59	1
0	8	14	0	37	1
1	8	14	326	355	26
2	8	14	280	292	37
3	8	14	189	142	57
4	8	14	0	168	1
5	8	14	310	264	35
6	8	14	525	475	22
7	8	14	0	114	1
8	8	14	0	8	1
9	8	14	171	138	91
10	8	14	0	151	1
1	9	14	0	108	1
2	9	14	168	90	67
3	9	14	264	325	40
4	9	14	78	12	77
5	9	14	681	619	19
6	9	14	464	469	24
7	9	14	274	294	46
8	9	14	0	110	1
9	9	14	0	143	1
0	10	14	185	196	115
1	10	14	0	107	1
2	10	14	0	70	1
3	10	14	0	89	1
4	10	14	0	263	1

Table 6. . Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
5	10	14	45	82	44
6	10	14	130	9	129
7	10	14	194	286	74
8	10	14	159	59	124
1	11	14	0	45	1
2	11	14	80	50	80
3	11	14	113	32	113
4	11	14	40	26	40
5	11	14	104	10	104
6	11	14	139	172	138
7	11	14	168	108	101
8	11	14	45	48	44
0	12	14	308	97	62
1	12	14	0	29	1
2	12	14	0	129	1
3	12	14	63	19	63
4	12	14	244	209	51
5	12	14	143	80	143
6	12	14	249	146	53
7	12	14	263	51	48
1	13	14	248	86	53
2	13	14	72	128	72
3	13	14	0	97	1
4	13	14	85	17	84
5	13	14	140	245	140
6	13	14	244	269	60
0	14	14	155	6	155
1	14	14	0	54	1
2	14	14	195	220	85
3	14	14	0	31	1
4	14	14	104	84	104
5	14	14	194	230	94
1	15	14	123	94	123
2	15	14	0	78	1
3	15	14	178	116	114
1	1	15	288	303	35
2	1	15	190	113	54
3	1	15	296	204	36
4	1	15	172	83	82
5	1	15	63	108	63
6	1	15	0	124	1
7	1	15	90	4	89
8	1	15	160	219	103
9	1	15	80	100	80
10	1	15	224	140	71
0	2	15	436	355	33
1	2	15	606	575	16
2	2	15	533	480	19
3	2	15	263	268	40
4	2	15	0	62	1
5	2	15	244	238	45
6	2	15	227	90	53
7	2	15	389	328	25
8	2	15	245	198	57
9	2	15	45	74	44
10	2	15	0	158	1
1	3	15	401	364	26
2	3	15	20	3	20
3	3	15	0	10	1
4	3	15	185	42	59
5	3	15	330	450	31
6	3	15	195	52	56
7	3	15	336	263	40
8	3	15	188	193	76
9	3	15	0	205	1
10	3	15	20	19	20
0	4	15	267	238	63
1	4	15	0	87	1
2	4	15	285	283	36
3	4	15	318	322	29
4	4	15	102	122	102
5	4	15	165	248	80
6	4	15	218	181	62
7	4	15	396	374	32
8	4	15	169	58	99
9	4	15	0	117	1
10	4	15	330	296	40
1	5	15	268	248	39
2	5	15	112	38	111
3	5	15	0	100	1
4	5	15	106	108	105
5	5	15	169	90	76
6	5	15	211	197	63
7	5	15	60	133	60
8	5	15	120	147	120
9	5	15	0	89	1
10	5	15	0	61	1
0	6	15	110	144	109
1	6	15	0	154	1
2	6	15	96	138	96
3	6	15	0	24	1
4	6	15	85	61	84
5	6	15	0	75	1
6	6	15	414	427	29
7	6	15	239	170	54
8	6	15	0	81	1
9	6	15	63	101	63
10	6	15	146	200	145
1	7	15	300	275	39
2	7	15	0	46	1
3	7	15	188	134	69
4	7	15	546	559	17
5	7	15	263	70	46
6	7	15	63	183	63
7	7	15	20	107	20
8	7	15	0	96	1
9	7	15	0	124	1
0	8	15	75	108	74
1	8	15	102	49	102
2	8	15	154	190	93
3	8	15	193	242	68
4	8	15	315	311	35
5	8	15	0	10	1
6	8	15	234	175	54
7	8	15	92	46	91
8	8	15	92	42	91
9	8	15	0	121	1
1	9	15	372	350	28
2	9	15	285	290	38
3	9	15	0	77	1
4	9	15	0	137	1
5	9	15	172	203	80
6	9	15	224	197	63
7	9	15	0	130	1
8	9	15	164	57	110
0	10	15	294	338	50
1	10	15	134	66	134
2	10	15	0	48	1
3	10	15	286	267	44
4	10	15	110	211	109
5	10	15	195	285	71
6	10	15	207	179	65
7	10	15	0	53	1
8	10	15	60	77	60
1	11	15	0	62	1
2	11	15	0	37	1
3	11	15	112	120	111
4	11	15	0	29	1
5	11	15	85	121	84
6	11	15	0	98	1
7	11	15	197	110	80
0	12	15	0	48	1
1	12	15	66	93	66
2	12	15	0	20	1
3	12	15	234	173	68
4	12	15	35	18	34
5	12	15	0	81	1
6	12	15	204	280	71
1	13	15	0	12	1
2	13	15	0	65	1
3	13	15	0	164	1
4	13	15	0	100	1
5	13	15	170	71	113
0	14	15	394	304	44
1	14	15	92	79	91
2	14	15	0	110	1
3	14	15	199	53	114
0	0	16	467	535	44
1	0	16	0	18	1
2	0	16	0	7	1
3	0	16	211	204	89
4	0	16	0	244	1
5	0	16	406	342	50
6	0	16	168	126	167
7	0	16	0	5	1
8	0	16	0	4	1
9	0	16	234	121	109
10	0	16	161	105	161
1	1	16	133	129	132
2	1	16	385	349	26
3	1	16	92	62	91
4	1	16	301	231	45
5	1	16	501	333	27
6	1	16	28	109	28
7	1	16	0	53	1
8	1	16	53	95	52
9	1	16	85	66	84
10	1	16	0	29	1
0	2	16	0	217	1
1	2	16	197	222	69
2	2	16	0	78	1
3	2	16	0	40	1
4	2	16	346	342	38
5	2	16	386	405	31
6	2	16	222	158	69
7	2	16	0	58	1
8	2	16	0	84	1
9	2	16	189	157	90
10	2	16	198	16	113
1	3	16	0	87	1
2	3	16	201	128	65
3	3	16	155	232	102
4	3	16	0	135	1
5	3	16	139	16	138
6	3	16	123	70	123
7	3	16	123	74	123
8	3	16	194	144	84
9	3	16	0	41	1
0	4	16	318	342	52
1	4	16	0	21	1
2	4	16	0	44	1
3	4	16	172	142	84
4	4	16	153	105	152
5	4	16	0	93	1
6	4	16	57	29	56
7	4	16	0	31	1
8	4	16	0	66	1
9	4	16	106	177	105
1	5	16	260	217	45
2	5	16	165	104	90
3	5	16	217	169	62
4	5	16	275	308	44
5	5	16	179	159	94
6	5	16	136	192	135
7	5	16	178	151	88
8	5	16	186	71	96
9	5	16	20	15	20
0	6	16	507	463	32
1	6	16	0	79	1
2	6	16	0	111	1
3	6	16	215	285	60
4	6	16	310	341	40
5	6	16	154	268	125
6	6	16	241	68	58
7	6	16	190	12	85
8	6	16	220	244	71
9	6	16	178	151	103
1	7	16	454	415	25
2	7	16	0	140	1
3	7	16	170	41	97
4	7	16	85	164	84
5	7	16	459	399	29
6	7	16	80	69	80
7	7	16	0	13	1
8	7	16	211	81	67
0	8	16	0	76	1
1	8	16	146	234	145
2	8	16	184	246	73
3	8	16	0	79	1
4	8	16	100	104	100
5	8	16	94	136	93
6	8	16	161	142	161
7	8	16	166	192	121
8	8	16	128	44	128
1	9	16	0	12	1
2	9	16	0	159	1
3	9	16	40	213	40
4	9	16	0	168	1
5	9	16	108	62	107
6	9	16	83	7	82
7	9	16	112	17	111
0	10	16	0	143	1
1	10	16	75	45	74
2	10	16	96	170	96
3	10	16	0	19	1
4	10	16	214	245	62
5	10	16	110	8	109
6	10	16	100	43	100
7	10	16	166	193	131
1	11	16	0	43	1
2	11	16	0	54	1
3	11	16	136	98	135
4	11	16	92	84	91
5	11	16	151	11	151
6	11	16	142	116	141
0	12	16	171	137	171
1	12	16	158	1	123
2	12	16	0	56	1
3	12	16	0	8	1
4	12	16	0	76	1
5	12	16	122	89	121
1	13	16	192	85	78
2	13	16	158	74	157
3	13	16	165	65	165
1	1	17	217	10	61
2	1	17	354	250	33
3	1	17	384	313	33
4	1	17	194	170	82
5	1	17	205	138	80
6	1	17	240	156	62
7	1	17	0	11	1
8	1	17	108	69	107
9	1	17	78	43	77
0	2	17	527	470	34
1	2	17	201	46	65
2	2	17	151	132	151
3	2	17	0	7	1
4	2	17	184	68	96
5	2	17	102	146	102
6	2	17	170	158	116
7	2	17	122	56	121
8	2	17	0	37	1
9	2	17	0	86	1
1	3	17	218	42	58
2	3	17	404	393	29
3	3	17	262	196	53
4	3	17	292	277	52
5	3	17	0	56	1
6	3	17	314	232	48
7	3	17	246	227	74
8	3	17	139	141	138
9	3	17	127	73	126
0	4	17	263	179	74
1	4	17	272	296	45
2	4	17	202	168	63
3	4	17	207	220	68
4	4	17	329	370	43
5	4	17	200	157	83
6	4	17	263	113	61
7	4	17	63	84	63
8	4	17	0	102	1
1	5	17	354	354	33
2	5	17	341	273	35
3	5	17	153	151	152
4	5	17	295	229	43
5	5	17	131	47	131
6	5	17	186	88	119
7	5	17	209	104	94
8	5	17	118	70	118
0	6	17	246	272	79
1	6	17	0	13	1
2	6	17	133	5	132
3	6	17	45	153	44
4	6	17	156	242	156
5	6	17	211	231	90
6	6	17	256	31	71
7	6	17	0	53	1
8	6	17	177	108	176
1	7	17	0	25	1
2	7	17	197	245	78
3	7	17	369	342	34
4	7	17	187	123	90
5	7	17	236	88	77
6	7	17	0	52	1
7	7	17	213	48	92
0	8	17	171	229	171
1	8	17	159	176	105
2	8	17	153	127	152
3	8	17	0	39	1
4	8	17	154	196	153
5	8	17	345	266	48
6	8	17	273	213	65
7	8	17	0	88	1
1	9	17	0	27	1
2	9	17	75	186	74
3	9	17	125	115	125
4	9	17	0	10	1
5	9	17	133	61	132
6	9	17	0	72	1
0	10	17	192	100	128
1	10	17	0	27	1
2	10	17	0	28	1
3	10	17	136	172	135
4	10	17	159	177	158
5	10	17	0	100	1
1	11	17	0	96	1
2	11	17	0	57	1
3	11	17	0	11	1
4	11	17	104	10	104
0	12	17	0	132	1
1	12	17	0	22	1
2	12	17	0	153	1
3	12	17	0	114	1
0	0	18	205	100	205
1	0	18	575	669	33
2	0	18	911	841	24
3	0	18	287	285	72
4	0	18	244	181	95
5	0	18	164	100	163
6	0	18	322	187	66
7	0	18	271	117	91
8	0	18	0	46	1
1	1	18	661	613	19
2	1	18	146	51	145
3	1	18	158	98	157
4	1	18	199	156	79
5	1	18	85	90	84
6	1	18	149	92	148
7	1	18	0	48	1
8	1	18	0	93	1
0	2	18	85	123	84
1	2	18	206	224	70
2	2	18	113	44	113
3	2	18	0	103	1
4	2	18	0	37	1
5	2	18	131	28	131
6	2	18	63	30	63
7	2	18	118	11	118
8	2	18	0	10	1
1	3	18	0	43	1
2	3	18	192	173	80
3	3	18	155	67	155
4	3	18	260	218	62
5	3	18	0	105	1
6	3	18	131	19	131
7	3	18	150	48	149
8	3	18	0	48	1
0	4	18	0	117	1
1	4	18	60	24	60
2	4	18	380	367	37
3	4	18	113	33	113
4	4	18	85	96	84
5	4	18	0	84	1
6	4	18	0	83	1
7	4	18	322	293	56
1	5	18	0	87	1
2	5	18	57	19	56
3	5	18	128	110	128
4	5	18	136	9	135
5	5	18	0	20	1
6	5	18	98	17	98
7	5	18	143	162	143
0	6	18	0	60	1
1	6	18	331	233	37
2	6	18	169	6	119
3	6	18	319	290	43
4	6	18	160	64	160
5	6	18	229	153	89
6	6	18	0	124	1
7	6	18	120	225	120
1	7	18	142	143	141
2	7	18	0	126	1
3	7	18	308	252	49
4	7	18	83	77	82
5	7	18	118	113	118
6	7	18	0	0	1
0	8	18	0	110	1
1	8	18	178	62	97
2	8	18	0	138	1
3	8	18	412	369	38
4	8	18	112	25	111
5	8	18	100	172	100
1	9	18	276	255	49
2	9	18	113	111	113
3	9	18	225	162	79

Table 6. Observed and calculated structure factors for af004a

h	k	l	10Fo	10Fc	10s
4	9	18	186	92	113
5	9	18	194	82	154
0	10	18	0	180	1
1	10	18	185	62	109
2	10	18	182	57	125
3	10	18	130	93	129
1	11	18	0	6	1
2	11	18	0	25	1
1	1	19	146	214	145
2	1	19	240	251	58
3	1	19	249	179	63
4	1	19	0	7	1
5	1	19	20	78	20
6	1	19	0	42	1
7	1	19	0	81	1
0	2	19	104	233	104
1	2	19	190	110	91
2	2	19	406	389	35
3	2	19	172	291	132
4	2	19	137	37	137
5	2	19	158	4	157
6	2	19	325	204	59
7	2	19	0	26	1
1	3	19	367	316	36
2	3	19	377	350	36
3	3	19	411	255	37
4	3	19	0	26	1
5	3	19	197	71	99
6	3	19	176	23	175
0	4	19	204	148	144
1	4	19	0	115	1
2	4	19	133	32	132
3	4	19	253	231	78
4	4	19	66	3	66
5	4	19	20	119	20
6	4	19	261	262	74
1	5	19	75	51	74
2	5	19	0	1	1
3	5	19	115	124	115
4	5	19	212	54	83
5	5	19	96	119	96
6	5	19	0	77	1
0	6	19	0	65	1
1	6	19	147	115	147
2	6	19	238	195	69
3	6	19	171	109	151
4	6	19	137	119	137
5	6	19	0	113	1
1	7	19	182	103	102
2	7	19	122	38	121
3	7	19	0	28	1
4	7	19	195	80	131
0	8	19	0	48	1
1	8	19	276	168	58
2	8	19	243	255	70
3	8	19	0	40	1
4	8	19	234	155	87
1	9	19	131	122	131
2	9	19	0	91	1
0	0	20	805	738	41
1	0	20	402	346	51
2	0	20	192	135	192
3	0	20	0	159	1
4	0	20	213	63	168
5	0	20	0	40	1
1	1	20	96	16	96
2	1	20	560	571	28
3	1	20	57	253	56
4	1	20	182	12	133
5	1	20	0	22	1
0	2	20	879	787	31
1	2	20	164	151	163
2	2	20	280	209	61
3	2	20	199	131	111
4	2	20	0	76	1
5	2	20	206	146	106
1	3	20	163	95	162
2	3	20	240	131	71
3	3	20	260	224	71
4	3	20	35	67	34
5	3	20	139	85	138
0	4	20	441	369	44
1	4	20	208	50	98
2	4	20	0	26	1
3	4	20	0	57	1
4	4	20	0	104	1
1	5	20	0	89	1
2	5	20	0	16	1
3	5	20	104	84	104
4	5	20	0	28	1
0	6	20	206	326	142
1	6	20	200	201	92
2	6	20	0	6	1
3	6	20	0	32	1
1	7	20	213	44	89
2	7	20	235	174	73
1	1	21	177	227	120
2	1	21	0	37	1
3	1	21	233	74	85
0	2	21	0	219	1
1	2	21	0	119	1
2	2	21	206	208	118
1	3	21	0	18	1
2	3	21	0	41	1
0	4	21	249	72	97
1	4	21	317	249	58

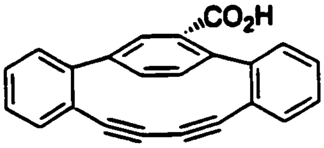

CO2H

Table 1. Crystal data and structure refinement for af006.

Identification code	af006
Empirical formula	$C_{23}H_{12}O_2$
Formula weight	320.33
Temperature	203(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 12.879(4) A alpha = 90 deg. b = 10.168(3) A beta = 114.545(4) de c = 13.466(4) A gamma = 90 deg.
Volume	1604.2(8) A^3
Z, Calculated density	4, 1.326 Mg/m^3
Absorption coefficient	0.084 mm^-1
F(000)	664
Crystal size	0.2 x 0.2 x 0.2 mm
Theta range for data collection	2.65 to 28.64 deg.
Limiting indices	-17<=h<=15, 0<=k<=12, 0<=l<=18
Reflections collected / unique	8740 / 3482 [R(int) = 0.1050]
Completeness to theta = 28.64	84.5 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3482 / 0 / 227
Goodness-of-fit on F^2	1.027
Final R indices [I>2sigma(I)]	R1 = 0.0563, wR2 = 0.0983
R indices (all data)	R1 = 0.1205, wR2 = 0.1047
Extinction coefficient	0.0127(11)
Largest diff. peak and hole	0.270 and -0.256 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af006. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	8726(1)	5731(2)	-654(1)	41(1)
O(2)	10078(1)	6190(2)	990(1)	41(1)
C(1)	9106(2)	6398(2)	210(2)	31(1)
C(2)	8401(2)	7494(2)	328(2)	27(1)
C(3)	7385(2)	7781(2)	-574(2)	32(1)
C(4)	6621(2)	8692(2)	-521(2)	32(1)
C(5)	6943(2)	9457(2)	410(2)	40(1)
C(6)	7948(2)	9188(2)	1311(2)	39(1)
C(7)	8659(2)	8170(2)	1309(2)	31(1)
C(8)	9471(2)	7659(2)	2399(2)	32(1)
C(9)	10541(2)	8165(3)	2998(2)	43(1)
C(10)	11221(2)	7687(3)	4032(2)	45(1)
C(11)	10822(2)	6676(3)	4453(2)	48(1)
C(12)	9752(2)	6163(2)	3880(2)	44(1)
C(13)	9061(2)	6642(2)	2852(2)	35(1)
C(14)	7908(2)	6203(2)	2230(2)	42(1)
C(15)	6954(2)	6159(3)	1540(2)	45(1)
C(16)	5959(2)	6405(3)	625(2)	46(1)
C(17)	5263(2)	6857(3)	-212(2)	46(1)
C(18)	4720(2)	7649(3)	-1177(2)	41(1)
C(19)	3589(2)	7520(3)	-1919(2)	49(1)
C(20)	3142(2)	8322(3)	-2806(2)	51(1)
C(21)	3806(2)	9272(3)	-2977(2)	56(1)
C(22)	4926(2)	9434(3)	-2251(2)	51(1)
C(23)	5410(2)	8633(2)	-1344(2)	35(1)

Table 3. Bond lengths [A] and angles [deg] for af006.

O(1)-C(1)	1.257(3)
O(2)-C(1)	1.273(2)
C(1)-C(2)	1.488(3)
C(2)-C(3)	1.396(3)
C(2)-C(7)	1.401(3)
C(3)-C(4)	1.375(3)
C(4)-C(5)	1.385(3)
C(4)-C(23)	1.492(3)
C(5)-C(6)	1.385(3)
C(6)-C(7)	1.383(3)
C(7)-C(8)	1.497(3)
C(8)-C(9)	1.375(3)
C(8)-C(13)	1.411(3)
C(9)-C(10)	1.389(3)
C(10)-C(11)	1.373(3)
C(11)-C(12)	1.372(3)
C(12)-C(13)	1.386(3)
C(13)-C(14)	1.439(3)
C(14)-C(15)	1.195(3)
C(15)-C(16)	1.382(4)
C(16)-C(17)	1.203(3)
C(17)-C(18)	1.439(3)
C(18)-C(19)	1.388(3)
C(18)-C(23)	1.417(3)
C(19)-C(20)	1.361(3)
C(20)-C(21)	1.372(3)
C(21)-C(22)	1.376(3)
C(22)-C(23)	1.382(3)

O(1)-C(1)-O(2)	123.0(2)
O(1)-C(1)-C(2)	118.9(2)
O(2)-C(1)-C(2)	118.1(2)
C(3)-C(2)-C(7)	119.1(2)
C(3)-C(2)-C(1)	117.2(2)
C(7)-C(2)-C(1)	123.5(2)
C(4)-C(3)-C(2)	121.9(2)
C(3)-C(4)-C(5)	118.2(2)
C(3)-C(4)-C(23)	119.0(2)
C(5)-C(4)-C(23)	121.5(2)
C(6)-C(5)-C(4)	120.2(2)
C(7)-C(6)-C(5)	121.6(2)
C(6)-C(7)-C(2)	118.1(2)
C(6)-C(7)-C(8)	116.8(2)
C(2)-C(7)-C(8)	123.3(2)
C(9)-C(8)-C(13)	119.0(2)
C(9)-C(8)-C(7)	124.6(2)
C(13)-C(8)-C(7)	116.3(2)
C(8)-C(9)-C(10)	121.2(2)
C(11)-C(10)-C(9)	119.2(2)
C(10)-C(11)-C(12)	120.8(3)
C(11)-C(12)-C(13)	120.4(2)
C(12)-C(13)-C(8)	119.3(2)
C(12)-C(13)-C(14)	123.4(2)
C(8)-C(13)-C(14)	117.2(2)
C(15)-C(14)-C(13)	160.6(3)
C(14)-C(15)-C(16)	163.7(3)

C(17)-C(16)-C(15)	163.5(3)
C(16)-C(17)-C(18)	162.5(3)
C(19)-C(18)-C(23)	119.5(3)
C(19)-C(18)-C(17)	124.6(3)
C(23)-C(18)-C(17)	115.9(2)
C(20)-C(19)-C(18)	120.7(3)
C(19)-C(20)-C(21)	120.2(2)
C(20)-C(21)-C(22)	120.5(3)
C(21)-C(22)-C(23)	120.8(3)
C(22)-C(23)-C(18)	118.3(2)
C(22)-C(23)-C(4)	125.5(2)
C(18)-C(23)-C(4)	116.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af006.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	40(1)	44(1)	30(1)	-13(1)	5(1)	4(1)
O(2)	39(1)	44(1)	35(1)	-6(1)	9(1)	14(1)
C(1)	25(1)	39(2)	26(2)	6(1)	6(1)	1(1)
C(2)	27(1)	26(2)	29(2)	2(1)	11(1)	0(1)
C(3)	35(1)	34(2)	26(2)	2(1)	12(1)	0(1)
C(4)	32(1)	29(2)	34(2)	6(1)	12(1)	2(1)
C(5)	41(2)	29(2)	47(2)	0(1)	15(1)	8(1)
C(6)	45(2)	33(2)	33(2)	-6(1)	12(1)	3(1)
C(7)	31(1)	29(2)	29(2)	1(1)	9(1)	-1(1)
C(8)	38(1)	28(2)	31(2)	-4(1)	14(1)	4(1)
C(9)	41(2)	47(2)	39(2)	-1(1)	15(1)	0(1)
C(10)	42(2)	56(2)	29(2)	-8(2)	7(1)	4(2)
C(11)	51(2)	55(2)	30(2)	1(2)	10(1)	13(2)
C(12)	52(2)	43(2)	34(2)	6(1)	16(1)	5(2)
C(13)	46(2)	33(2)	26(2)	-2(1)	14(1)	4(1)
C(14)	51(2)	38(2)	38(2)	8(1)	19(1)	-1(2)
C(15)	51(2)	41(2)	44(2)	9(2)	21(2)	-6(2)
C(16)	49(2)	44(2)	47(2)	3(2)	19(2)	-18(2)
C(17)	42(2)	45(2)	49(2)	-2(2)	17(1)	-15(2)
C(18)	31(1)	44(2)	42(2)	-3(2)	9(1)	3(1)
C(19)	38(2)	48(2)	58(2)	-8(2)	17(1)	-1(2)
C(20)	32(2)	62(2)	43(2)	-12(2)	0(1)	6(2)
C(21)	43(2)	64(2)	48(2)	7(2)	6(2)	16(2)
C(22)	40(2)	57(2)	51(2)	14(2)	13(1)	4(2)
C(23)	33(1)	33(2)	35(2)	-2(1)	11(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af006.

	x	y	z	U(eq)
H(2A)	10399	5574	826	62
H(3A)	7220	7339	-1235	38
H(5A)	6479	10161	430	48
H(6A)	8151	9710	1941	46
H(9A)	10818	8847	2702	51
H(10A)	11945	8052	4439	54
H(11A)	11287	6331	5144	57
H(12A)	9487	5481	4185	53
H(19A)	3127	6873	-1807	59
H(20A)	2376	8223	-3304	61
H(21A)	3493	9818	-3595	68
H(22A)	5368	10097	-2373	62

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
3	0	0	68	77	6
4	0	0	173	170	4
5	0	0	600	602	11
6	0	0	316	323	6
7	0	0	91	88	6
8	0	0	102	100	6
9	0	0	101	96	7
10	0	0	98	92	8
11	0	0	41	35	29
12	0	0	76	73	21
13	0	0	78	85	23
14	0	0	13	16	12
1	1	0	841	834	12
2	1	0	436	463	8
3	1	0	45	51	6
4	1	0	238	248	3
5	1	0	133	137	3
6	1	0	194	200	4
7	1	0	11	7	11
8	1	0	87	88	5
9	1	0	32	26	20
10	1	0	19	22	19
11	1	0	38	44	23
12	1	0	0	6	1
13	1	0	45	34	27
14	1	0	22	14	22
15	1	0	7	32	6
0	2	0	1198	1134	20
1	2	0	351	361	4
2	2	0	218	215	5
3	2	0	147	152	3
4	2	0	108	117	3
5	2	0	288	290	4
6	2	0	112	117	3
7	2	0	39	37	10
8	2	0	16	14	15
9	2	0	62	67	10
10	2	0	212	221	5
11	2	0	47	53	17
12	2	0	11	19	10
13	2	0	14	12	14
14	2	0	38	36	37
15	2	0	30	36	30
1	3	0	713	673	8
2	3	0	0	9	1
3	3	0	150	135	4
4	3	0	51	46	5
5	3	0	153	152	3
6	3	0	172	174	3
7	3	0	83	90	5
8	3	0	270	280	5
9	3	0	97	90	7
10	3	0	181	180	7
11	3	0	187	177	7
12	3	0	22	29	21
13	3	0	64	54	19
14	3	0	22	24	21
15	3	0	37	5	36
0	4	0	606	618	11
1	4	0	273	275	4
2	4	0	209	207	3
3	4	0	398	366	7
4	4	0	73	72	6
5	4	0	144	138	5
6	4	0	12	6	12
7	4	0	23	16	23
8	4	0	101	97	9
9	4	0	279	279	8
10	4	0	240	235	7
11	4	0	37	33	36
12	4	0	26	34	25
13	4	0	78	71	16
14	4	0	14	7	14
1	5	0	9	6	9
2	5	0	427	401	5
3	5	0	404	389	5
4	5	0	224	222	5
5	5	0	9	22	9
6	5	0	53	52	16
7	5	0	108	107	8
8	5	0	305	307	8
9	5	0	85	94	12
10	5	0	65	63	16
11	5	0	108	108	10
12	5	0	43	46	43
13	5	0	64	58	22
14	5	0	46	22	45
0	6	0	247	238	6
1	6	0	0	11	1
2	6	0	156	161	3
3	6	0	65	60	11
4	6	0	36	35	35
5	6	0	174	171	6
6	6	0	190	197	7
7	6	0	43	41	29
8	6	0	151	150	8
9	6	0	52	70	22
10	6	0	24	37	24
11	6	0	18	9	18
12	6	0	95	78	14
13	6	0	46	37	46
1	7	0	162	168	6
2	7	0	62	64	12
3	7	0	86	93	10
4	7	0	114	117	7
5	7	0	96	104	9
6	7	0	189	187	7
7	7	0	110	123	9
8	7	0	103	94	10
9	7	0	126	121	9
10	7	0	26	18	25
11	7	0	84	74	14
12	7	0	29	33	28
1	8	0	27	24	27
2	8	0	41	46	23
3	8	0	164	171	6
4	8	0	39	57	32
5	8	0	173	179	7
6	8	0	53	72	19
7	8	0	0	8	1
8	8	0	10	27	10
9	8	0	72	62	15
10	8	0	60	57	21
11	8	0	111	106	12
2	9	0	57	56	24
3	9	0	77	70	16
4	9	0	71	68	13
5	9	0	59	56	17
6	9	0	59	63	18
7	9	0	24	36	24
8	9	0	41	32	41
9	9	0	104	92	11
10	9	0	68	60	20
11	9	0	85	81	25
4	10	0	87	108	17
5	10	0	74	90	21
6	10	0	0	5	1
7	10	0	45	27	38
8	10	0	43	45	43
9	10	0	0	27	1
10	10	0	0	11	1
6	11	0	17	38	16
7	11	0	0	17	1
8	11	0	0	36	1
9	11	0	0	29	1
-16	1	1	19	15	18
-15	1	1	13	1	13
-14	1	1	32	8	31
-13	1	1	15	16	15
-12	1	1	15	34	15
-11	1	1	55	56	11
-10	1	1	79	73	7
-9	1	1	77	74	6
-8	1	1	229	234	5
-7	1	1	86	94	4
-6	1	1	69	77	5
-5	1	1	186	185	3
-4	1	1	293	308	4
-3	1	1	103	112	3
-2	1	1	198	210	3
1	1	1	114	111	2
2	1	1	357	358	4
3	1	1	54	50	5
4	1	1	83	88	3
5	1	1	304	313	7
6	1	1	80	80	5
7	1	1	181	186	4
8	1	1	136	138	4
9	1	1	50	52	10
10	1	1	67	71	9
11	1	1	58	43	13
12	1	1	37	51	36
13	1	1	27	30	27
14	1	1	0	16	1
15	1	1	20	13	19
-15	2	1	0	5	1
-14	2	1	43	30	42
-13	2	1	119	115	10
-12	2	1	0	2	1
-11	2	1	95	96	8
-10	2	1	49	46	15
-9	2	1	47	49	11
-8	2	1	39	56	12
-7	2	1	344	349	5
-6	2	1	65	74	5
-5	2	1	16	5	15
-4	2	1	149	146	3
-3	2	1	808	803	10
-2	2	1	489	476	13
-1	2	1	499	532	6
0	2	1	1006	1038	12
1	2	1	235	239	5
2	2	1	63	53	4
3	2	1	25	22	10
4	2	1	319	333	4
5	2	1	5	1	4
6	2	1	287	298	4
7	2	1	223	229	4
8	2	1	76	76	6
9	2	1	198	209	5
10	2	1	258	267	11
11	2	1	64	69	14
12	2	1	122	115	9
13	2	1	58	51	22
14	2	1	29	44	29
-15	3	1	51	44	50
-14	3	1	17	2	17
-13	3	1	52	39	30
-12	3	1	0	20	1
-11	3	1	22	36	22
-10	3	1	232	223	6
-9	3	1	31	17	30
-8	3	1	20	16	19
-7	3	1	51	50	8
-6	3	1	86	81	5
-5	3	1	63	64	5
-4	3	1	149	142	3
-3	3	1	75	79	6
-2	3	1	38	40	8
-1	3	1	414	420	5
0	3	1	144	141	3
1	3	1	113	126	3
2	3	1	155	157	5
3	3	1	293	273	5
4	3	1	140	145	3
5	3	1	218	218	3
6	3	1	104	102	4
7	3	1	0	4	1
8	3	1	115	114	6
9	3	1	77	79	12
10	3	1	101	103	9
11	3	1	10	4	9
12	3	1	35	44	34
13	3	1	31	47	31
14	3	1	0	11	1
-15	4	1	43	42	42
-14	4	1	26	20	25
-13	4	1	113	97	12
-12	4	1	0	11	1
-11	4	1	137	129	8
-10	4	1	25	43	25
-9	4	1	0	2	1
-8	4	1	237	224	7
-7	4	1	197	189	5
-6	4	1	52	53	8
-5	4	1	122	116	6
-4	4	1	33	41	18
-3	4	1	40	23	8
-2	4	1	636	616	8
-1	4	1	64	60	5
0	4	1	63	68	5
1	4	1	57	56	4
2	4	1	21	42	20
3	4	1	72	72	5
4	4	1	46	43	11
5	4	1	277	275	5
6	4	1	203	194	6
7	4	1	82	94	11
8	4	1	17	9	17
9	4	1	302	302	8
10	4	1	178	174	7
11	4	1	31	26	31
12	4	1	70	68	16
13	4	1	27	29	27
14	4	1	98	90	15
-14	5	1	0	13	1
-13	5	1	39	30	38
-12	5	1	70	68	18
-11	5	1	0	20	1
-10	5	1	23	6	23
-9	5	1	20	6	20
-8	5	1	145	147	12
-7	5	1	23	7	23
-6	5	1	11	20	10
-5	5	1	45	54	14
-4	5	1	123	123	3
-3	5	1	111	120	3
-2	5	1	70	64	5
-1	5	1	118	119	3
0	5	1	160	162	3
1	5	1	300	299	4
2	5	1	107	105	4
3	5	1	23	14	22
4	5	1	65	66	11
5	5	1	26	37	25
6	5	1	154	158	7
7	5	1	249	245	7
8	5	1	137	133	8
9	5	1	17	22	17
10	5	1	92	88	12
11	5	1	48	55	28
12	5	1	22	12	21
13	5	1	0	29	1
-13	6	1	12	15	11
-12	6	1	110	104	12
-11	6	1	36	35	35
-10	6	1	27	34	26
-9	6	1	54	71	20
-8	6	1	0	11	1
-7	6	1	118	121	9
-6	6	1	23	23	23
-5	6	1	61	61	13
-4	6	1	104	114	7
-3	6	1	45	50	13
-2	6	1	98	93	4
-1	6	1	454	445	6
0	6	1	422	416	5
1	6	1	64	65	6
2	6	1	41	39	20
3	6	1	184	188	6
4	6	1	64	73	12
5	6	1	75	69	12
6	6	1	74	78	12
7	6	1	245	244	7
8	6	1	168	168	7
9	6	1	248	239	8
10	6	1	8	16	7
11	6	1	16	18	16
12	6	1	30	32	30
13	6	1	66	46	36
-13	7	1	67	39	24
-12	7	1	101	83	14
-11	7	1	65	51	19
-10	7	1	137	129	9
-9	7	1	62	59	16
-8	7	1	37	32	36
-7	7	1	46	33	24
-6	7	1	106	109	9
-5	7	1	0	30	1
-4	7	1	235	223	7
-3	7	1	53	48	15
-2	7	1	75	74	9
-1	7	1	216	219	4
0	7	1	49	46	10
1	7	1	161	154	6
2	7	1	78	87	10
3	7	1	213	216	7
4	7	1	68	71	13
5	7	1	87	97	11
6	7	1	66	71	14
7	7	1	9	42	9
8	7	1	117	129	9
9	7	1	49	39	26
10	7	1	54	62	23
11	7	1	82	88	14
12	7	1	22	14	22
-12	8	1	38	41	37
-11	8	1	75	70	18
-10	8	1	19	6	19
-9	8	1	10	9	10
-8	8	1	37	44	37
-7	8	1	101	155	15
-6	8	1	35	24	35
-5	8	1	10	0	9
-4	8	1	76	78	12
-3	8	1	0	24	1
-2	8	1	93	95	13
-1	8	1	115	117	9
0	8	1	117	117	10
1	8	1	31	19	31
2	8	1	19	16	18
3	8	1	75	68	11
4	8	1	68	64	14
5	8	1	142	141	8
6	8	1	122	117	8
7	8	1	35	43	35
8	8	1	47	60	30
9	8	1	53	67	25
10	8	1	50	42	27
11	8	1	0	4	1
-11	9	1	50	56	49
-10	9	1	53	47	30
-9	9	1	46	27	45
-8	9	1	45	38	32
-7	9	1	0	10	1
-6	9	1	73	93	14
-5	9	1	45	38	27
-4	9	1	0	13	1
-3	9	1	132	134	10
2	9	1	78	71	16
3	9	1	50	50	21
4	9	1	64	69	15
5	9	1	95	100	10
6	9	1	33	26	33
7	9	1	36	26	36
8	9	1	15	3	14
9	9	1	0	7	1
10	9	1	153	137	9
-10	10	1	36	31	36
-9	10	1	45	33	45
-8	10	1	0	11	1
-7	10	1	19	24	18
-6	10	1	102	110	16
-5	10	1	38	25	38
3	10	1	36	7	36
4	10	1	87	98	16
5	10	1	76	89	14
6	10	1	0	13	1
7	10	1	26	47	25
8	10	1	28	4	28
9	10	1	146	134	9
-9	11	1	0	2	1
-8	11	1	60	56	47
-7	11	1	24	38	23
-6	11	1	0	5	1
5	11	1	0	8	1
6	11	1	109	111	16
7	11	1	33	33	33
8	11	1	29	11	29
-8	12	1	55	55	55
7	12	1	45	26	44
-16	0	2	0	20	1
-15	0	2	62	18	45
-14	0	2	20	17	20
-13	0	2	151	139	8
-12	0	2	309	294	7
-11	0	2	57	59	16
-10	0	2	56	47	15
-9	0	2	355	347	7
-8	0	2	262	249	6
-7	0	2	459	458	9
-6	0	2	22	34	21
-5	0	2	38	59	11
-4	0	2	71	75	6
-3	0	2	346	357	6
-2	0	2	448	459	10
0	0	2	201	205	6
2	0	2	241	246	5
3	0	2	201	192	5
4	0	2	339	329	7
5	0	2	312	314	6
6	0	2	0	17	1
7	0	2	323	329	7
8	0	2	63	61	11
9	0	2	79	67	9

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
10	0	2	52	62	16
11	0	2	93	114	16
12	0	2	28	4	28
-16	1	2	44	27	44
-15	1	2	0	13	1
-14	1	2	66	75	15
-13	1	2	162	156	6
-12	1	2	0	5	1
-11	1	2	178	176	4
-10	1	2	109	108	5
-9	1	2	111	108	5
-8	1	2	16	20	16
-7	1	2	323	314	5
-6	1	2	8	16	7
-5	1	2	223	231	3
-4	1	2	23	20	23
-3	1	2	235	233	3
-2	1	2	1082	1200	34
-1	1	2	806	817	11
0	1	2	472	487	7
1	1	2	374	351	6
2	1	2	150	142	3
3	1	2	135	140	3
4	1	2	70	76	5
5	1	2	551	546	8
6	1	2	210	219	4
7	1	2	99	95	5
8	1	2	96	96	5
9	1	2	118	121	5
10	1	2	188	197	5
11	1	2	33	42	33
12	1	2	83	76	21
13	1	2	0	11	1
14	1	2	64	44	63
-15	2	2	25	2	25
-14	2	2	26	18	26
-13	2	2	38	63	37
-12	2	2	162	162	6
-11	2	2	72	62	10
-10	2	2	23	23	23
-9	2	2	275	279	4
-8	2	2	194	192	4
-7	2	2	80	77	5
-6	2	2	138	132	3
-5	2	2	337	327	4
-4	2	2	226	218	3
-3	2	2	1561	1473	19
-2	2	2	306	316	5
-1	2	2	576	624	41
0	2	2	49	51	6
1	2	2	172	171	4
2	2	2	328	338	4
3	2	2	47	49	6
4	2	2	125	123	3
5	2	2	132	135	3
6	2	2	33	42	13
7	2	2	0	7	1
8	2	2	135	139	5
9	2	2	72	83	10
10	2	2	51	47	18
11	2	2	82	85	12
12	2	2	46	52	30
13	2	2	24	28	24
14	2	2	47	39	46
-16	3	2	52	21	51
-15	3	2	0	21	1
-14	3	2	20	27	19
-13	3	2	32	34	31
-12	3	2	106	97	11
-11	3	2	164	161	6
-10	3	2	253	238	6
-9	3	2	13	23	12
-8	3	2	9	6	9
-7	3	2	168	159	4
-6	3	2	87	73	4
-5	3	2	94	100	4
-4	3	2	531	527	6
-3	3	2	752	740	11
-2	3	2	231	236	3
-1	3	2	184	180	3
0	3	2	543	630	21
1	3	2	215	210	4
2	3	2	204	199	4
3	3	2	162	171	3
4	3	2	59	55	4
5	3	2	113	123	3
6	3	2	65	67	8
7	3	2	0	0	1
8	3	2	52	53	18
9	3	2	47	44	20
10	3	2	72	78	13
11	3	2	0	0	1
12	3	2	0	19	1
13	3	2	36	32	36
14	3	2	31	33	31
-15	4	2	43	37	42
-14	4	2	49	36	41
-13	4	2	0	5	1
-12	4	2	120	96	11
-11	4	2	74	65	14
-10	4	2	10	6	9
-9	4	2	329	311	8
-8	4	2	66	72	10
-7	4	2	97	93	7
-6	4	2	143	142	4
-5	4	2	35	36	19
-4	4	2	165	158	4
-3	4	2	716	708	9
-2	4	2	487	493	6
-1	4	2	87	87	3
0	4	2	83	89	3
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2	4	2	377	379	7
3	4	2	52	54	13
4	4	2	84	91	9
5	4	2	54	55	14
6	4	2	21	32	21
7	4	2	290	291	7
8	4	2	94	88	10
9	4	2	197	200	7
10	4	2	37	41	36
11	4	2	35	26	35
12	4	2	8	3	7
13	4	2	51	54	33
14	4	2	0	3	1
-14	5	2	64	43	25
-13	5	2	24	22	24
-12	5	2	33	29	33
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-10	5	2	92	97	12
-9	5	2	23	34	22
-8	5	2	195	186	11
-7	5	2	230	217	5
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-5	5	2	87	91	7
-4	5	2	66	58	6
-3	5	2	57	57	6
-2	5	2	112	113	4
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0	5	2	160	170	3
1	5	2	202	204	4
2	5	2	93	109	12
3	5	2	133	130	6
4	5	2	162	158	6
5	5	2	198	205	6
6	5	2	40	46	36
7	5	2	42	42	31
8	5	2	225	219	7
9	5	2	70	80	14
10	5	2	13	16	12
11	5	2	0	16	1
12	5	2	40	8	40
13	5	2	47	53	46
-14	6	2	78	56	31
-13	6	2	0	6	1
-12	6	2	10	16	9
-11	6	2	123	114	10
-10	6	2	45	45	34
-9	6	2	70	60	14
-8	6	2	150	145	8
-7	6	2	54	40	17
-6	6	2	65	84	12
-5	6	2	129	123	7
-4	6	2	65	47	9
-3	6	2	37	41	13
-2	6	2	60	52	7
-1	6	2	132	134	4
0	6	2	30	28	17
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2	6	2	288	308	6
3	6	2	153	179	13
4	6	2	116	125	7
5	6	2	65	79	12
6	6	2	39	52	38
7	6	2	324	344	8
8	6	2	144	147	12
9	6	2	0	25	1
10	6	2	56	43	20
11	6	2	43	0	43
12	6	2	0	11	1
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-12	7	2	191	158	9
-11	7	2	91	64	14
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-9	7	2	11	9	10
-8	7	2	103	87	10
-7	7	2	191	208	21
-6	7	2	0	8	1
-5	7	2	104	106	9
-4	7	2	78	76	10
-3	7	2	123	119	7
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0	7	2	12	15	12
1	7	2	115	120	7
2	7	2	34	41	34
3	7	2	0	11	1
4	7	2	111	122	8
5	7	2	221	236	7
6	7	2	24	35	24
7	7	2	57	71	17
8	7	2	35	46	35
9	7	2	68	69	15
10	7	2	103	118	11
11	7	2	108	112	12
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-11	8	2	15	4	15
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-9	8	2	25	22	25
-8	8	2	33	60	32
-7	8	2	107	110	9
-6	8	2	60	58	16
-5	8	2	110	115	8
-4	8	2	25	23	24
-3	8	2	24	14	24
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0	8	2	61	53	12
1	8	2	36	51	36
2	8	2	20	19	19
3	8	2	51	45	17
4	8	2	93	97	11
5	8	2	43	52	29
6	8	2	32	13	31
7	8	2	60	72	17
8	8	2	37	22	36
9	8	2	70	68	16
10	8	2	42	53	41
-11	9	2	133	108	11
-10	9	2	22	34	21
-9	9	2	90	112	15
-8	9	2	31	4	30
-7	9	2	0	21	1
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-5	9	2	110	106	14
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1	9	2	42	60	42
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5	9	2	101	106	11
6	9	2	37	45	36
7	9	2	71	76	15
8	9	2	47	17	34
9	9	2	0	20	1
10	9	2	39	43	38
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-10	10	2	33	28	32
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-8	10	2	82	87	15
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5	10	2	0	6	1
6	10	2	0	7	1
7	10	2	39	50	39
8	10	2	46	35	45
9	10	2	30	0	29
-9	11	2	20	28	19
-8	11	2	0	10	1
-7	11	2	0	32	1
4	11	2	56	69	31
5	11	2	125	121	13
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6	12	2	64	44	38
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-15	1	3	53	36	31
-14	1	3	0	6	1
-13	1	3	148	131	6
-12	1	3	16	2	15
-11	1	3	111	107	6
-10	1	3	58	61	8
-9	1	3	0	9	1
-8	1	3	35	32	13
-7	1	3	105	101	4
-6	1	3	216	217	3
-5	1	3	34	27	9
-4	1	3	39	35	8
-3	1	3	803	855	17
-2	1	3	1072	1180	86
-1	1	3	355	350	7
0	1	3	13	7	13
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2	1	3	197	207	3
3	1	3	558	548	7
4	1	3	48	55	8
5	1	3	13	22	13
6	1	3	0	11	1
7	1	3	0	8	1
8	1	3	103	105	5
9	1	3	51	59	15
10	1	3	62	63	12
11	1	3	0	3	1
12	1	3	11	8	10
13	1	3	40	34	40
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-15	2	3	66	42	22
-14	2	3	0	1	1
-13	2	3	51	43	19
-12	2	3	172	152	6
-11	2	3	212	199	6
-10	2	3	150	149	4
-9	2	3	58	56	9
-8	2	3	167	160	4
-7	2	3	0	9	1
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-5	2	3	556	537	7
-4	2	3	260	276	4
-3	2	3	1695	1639	27
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0	2	3	134	141	3
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2	2	3	196	191	3
3	2	3	0	2	1
4	2	3	301	299	4
5	2	3	78	78	5
6	2	3	34	36	16
7	2	3	95	110	6
8	2	3	33	31	32
9	2	3	47	53	20
10	2	3	20	23	19
11	2	3	53	56	21
12	2	3	114	113	10
13	2	3	27	7	27
14	2	3	49	42	48
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-14	3	3	0	17	1
-13	3	3	26	26	26
-12	3	3	12	35	11
-11	3	3	122	125	7
-10	3	3	66	74	11
-9	3	3	53	52	11
-8	3	3	8	6	7
-7	3	3	25	33	24
-6	3	3	0	15	1
-5	3	3	69	74	5
-4	3	3	184	191	7
-3	3	3	192	189	4
-2	3	3	876	884	13
-1	3	3	337	344	12
0	3	3	91	96	3
1	3	3	47	44	6
2	3	3	146	151	3
3	3	3	371	378	5
4	3	3	45	42	7
5	3	3	139	140	3
6	3	3	131	142	7
7	3	3	85	86	10
8	3	3	129	126	8
9	3	3	3	12	3
10	3	3	74	78	13
11	3	3	28	44	27
12	3	3	35	28	34
13	3	3	62	61	23
-15	4	3	36	24	36
-14	4	3	38	21	37
-13	4	3	0	17	1
-12	4	3	120	109	10
-11	4	3	31	29	31
-10	4	3	21	6	20
-9	4	3	178	159	6
-8	4	3	34	40	34
-7	4	3	12	11	11
-6	4	3	342	331	5
-5	4	3	84	79	8
-4	4	3	323	301	6
-3	4	3	150	139	3
-2	4	3	167	164	3
-1	4	3	74	68	4
0	4	3	390	391	5
1	4	3	219	207	4
2	4	3	60	67	10
3	4	3	262	263	4
4	4	3	189	176	10
5	4	3	122	127	7
6	4	3	98	103	10
7	4	3	181	184	7
8	4	3	71	55	14
9	4	3	37	47	37
10	4	3	0	3	1
11	4	3	24	21	23
12	4	3	68	52	19
13	4	3	44	40	43
-15	5	3	30	38	30
-14	5	3	51	43	51
-13	5	3	94	85	14
-12	5	3	43	39	42
-11	5	3	44	29	44
-10	5	3	70	56	17
-9	5	3	60	59	18
-8	5	3	23	12	23
-7	5	3	105	101	7
-6	5	3	148	149	7
-5	5	3	37	39	20
-4	5	3	44	48	10
-3	5	3	225	222	4
-2	5	3	178	172	3
-1	5	3	265	271	4
0	5	3	52	61	8
1	5	3	52	44	9
2	5	3	154	158	6
3	5	3	223	219	6
4	5	3	90	84	10
5	5	3	65	65	14
6	5	3	122	126	8
7	5	3	19	22	19
8	5	3	85	82	11
9	5	3	0	19	1
10	5	3	79	85	13
11	5	3	32	4	32
12	5	3	30	15	29
-14	6	3	92	72	18
-13	6	3	108	97	13
-12	6	3	110	86	12
-11	6	3	29	16	28
-10	6	3	58	47	23
-9	6	3	82	92	13
-8	6	3	111	105	10
-7	6	3	114	125	8
-6	6	3	169	166	6
-5	6	3	125	123	6
-4	6	3	90	94	5
-3	6	3	188	181	4
-2	6	3	185	183	4
-1	6	3	89	78	4
0	6	3	348	354	5
1	6	3	47	53	17
2	6	3	187	207	14
3	6	3	77	69	11
4	6	3	59	58	15

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
5	6	3	149	150	7
6	6	3	189	196	7
7	6	3	124	130	9
8	6	3	79	96	12
9	6	3	76	80	14
10	6	3	117	122	9
11	6	3	57	24	25
12	6	3	31	19	31
-13	7	3	44	31	43
-12	7	3	84	54	18
-11	7	3	65	62	24
-10	7	3	72	60	18
-9	7	3	0	11	1
-8	7	3	44	53	30
-7	7	3	0	21	1
-6	7	3	48	44	22
-5	7	3	90	91	10
-4	7	3	26	16	26
-3	7	3	11	1	10
-2	7	3	142	147	5
-1	7	3	71	74	8
0	7	3	25	18	24
1	7	3	58	60	13
2	7	3	20	21	20
3	7	3	80	99	11
4	7	3	92	96	10
5	7	3	106	113	9
6	7	3	212	227	7
7	7	3	100	100	11
8	7	3	33	38	33
9	7	3	50	47	24
10	7	3	52	57	27
11	7	3	73	83	18
-12	8	3	52	26	42
-11	8	3	24	26	24
-10	8	3	42	32	42
-9	8	3	34	27	34
-8	8	3	50	45	26
-7	8	3	31	53	31
-6	8	3	131	124	8
-5	8	3	127	131	8
-4	8	3	129	118	11
-3	8	3	57	31	26
-1	8	3	44	49	22
0	8	3	0	10	1
1	8	3	61	74	13
2	8	3	23	20	22
3	8	3	149	155	7
4	8	3	164	176	8
5	8	3	22	16	21
6	8	3	50	60	27
7	8	3	25	22	25
8	8	3	42	47	42
9	8	3	45	55	33
10	8	3	47	45	46
-12	9	3	41	27	40
-11	9	3	0	13	1
-10	9	3	26	27	25
-9	9	3	75	72	20
-8	9	3	106	98	12
-7	9	3	19	8	19
-6	9	3	48	37	48
-5	9	3	0	15	1
-4	9	3	148	148	11
0	9	3	57	62	22
1	9	3	99	108	9
2	9	3	151	159	7
3	9	3	143	150	7
4	9	3	140	149	8
5	9	3	51	43	24
6	9	3	35	42	35
7	9	3	53	53	24
8	9	3	0	22	1
9	9	3	0	11	1
-11	10	3	0	22	1
-10	10	3	22	6	21
-9	10	3	37	14	36
-8	10	3	103	87	20
-7	10	3	0	30	1
-6	10	3	87	82	20
2	10	3	60	67	27
3	10	3	37	40	36
4	10	3	22	36	22
5	10	3	43	48	42
6	10	3	42	38	42
7	10	3	49	43	32
8	10	3	22	8	22
-9	11	3	67	60	46
-8	11	3	9	1	9
4	11	3	52	24	51
5	11	3	0	16	1
6	11	3	38	41	37
7	11	3	53	67	32
-16	0	4	0	26	1
-15	0	4	113	103	17
-14	0	4	201	187	8
-13	0	4	113	108	9
-12	0	4	255	249	7
-11	0	4	66	67	12
-10	0	4	139	141	6
-9	0	4	91	104	7
-8	0	4	214	245	25
-7	0	4	228	236	5
-6	0	4	92	88	6
-5	0	4	252	271	5
-4	0	4	105	115	5
-3	0	4	376	383	7
-2	0	4	61	62	9
-1	0	4	14	6	13
0	0	4	62	70	14
1	0	4	252	227	9
2	0	4	183	179	5
3	0	4	248	243	6
4	0	4	270	265	6
5	0	4	306	305	7
6	0	4	226	226	6
7	0	4	408	423	8
8	0	4	158	153	7
9	0	4	383	403	8
10	0	4	63	59	29
11	0	4	0	0	1
-16	1	4	89	85	17
-15	1	4	30	33	30
-14	1	4	48	31	21
-13	1	4	153	145	6
-12	1	4	55	65	11
-11	1	4	47	52	13
-10	1	4	113	120	5
-9	1	4	75	75	6
-8	1	4	190	188	4
-7	1	4	10	19	10
-6	1	4	49	46	7
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6	10	4	46	33	46
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Table 6. Observed and calculated structure factors for af006

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3	3	6	303	313	5
4	3	6	156	156	7
5	3	6	81	95	11
6	3	6	238	241	7
7	3	6	101	94	10
8	3	6	0	5	1
9	3	6	21	13	21
10	3	6	52	56	32
11	3	6	89	80	16
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-12	4	6	93	94	9
-11	4	6	151	153	6
-10	4	6	30	32	29
-9	4	6	120	116	5
-8	4	6	95	98	6
-7	4	6	12	30	11
-6	4	6	151	154	5
-5	4	6	744	763	13
-4	4	6	178	173	5
-3	4	6	259	250	5
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-1	4	6	70	69	7
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1	4	6	147	139	7
2	4	6	33	31	33
3	4	6	13	9	13
4	4	6	91	101	10
5	4	6	89	90	11
6	4	6	130	140	9
7	4	6	68	66	15
8	4	6	58	75	20
9	4	6	0	2	1
10	4	6	39	49	39
11	4	6	67	67	23
-15	5	6	36	11	36
-14	5	6	69	55	22
-13	5	6	124	112	11
-12	5	6	33	12	32
-11	5	6	24	7	23
-10	5	6	5	4	4
-9	5	6	101	90	8
-8	5	6	60	62	13
-7	5	6	32	24	32
-6	5	6	90	84	7
-5	5	6	215	207	5
-4	5	6	310	307	5
-3	5	6	19	10	19
-2	5	6	25	9	25
-1	5	6	100	93	6
0	5	6	86	83	7
1	5	6	197	192	7
2	5	6	73	72	12
3	5	6	107	108	9
4	5	6	36	39	35
5	5	6	20	12	19
6	5	6	28	23	27
7	5	6	48	38	27
8	5	6	45	45	35
9	5	6	24	34	24
10	5	6	59	57	24
-14	6	6	41	37	41
-13	6	6	18	2	17
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-11	6	6	108	98	12
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-8	6	6	146	134	7
-7	6	6	126	119	7
-6	6	6	76	78	8
-5	6	6	288	284	5
-4	6	6	124	121	5
-3	6	6	159	157	4
-2	6	6	126	124	6
-1	6	6	134	126	8
0	6	6	135	124	11
1	6	6	116	100	9
2	6	6	131	124	8
3	6	6	45	54	24
4	6	6	0	13	1
5	6	6	28	49	27
6	6	6	54	40	22
7	6	6	16	45	15
8	6	6	0	13	1
9	6	6	0	3	1
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-11	7	6	0	13	1
-10	7	6	62	50	21
-9	7	6	133	128	9
-8	7	6	69	65	16
-7	7	6	73	71	12
-6	7	6	118	125	7
-5	7	6	70	82	11
-4	7	6	47	62	18
-3	7	6	324	316	6
-2	7	6	228	224	7
-1	7	6	115	131	8
0	7	6	202	204	7
1	7	6	156	159	7
2	7	6	151	154	8
3	7	6	192	208	7
4	7	6	22	18	21
5	7	6	13	22	12
6	7	6	5	15	4
7	7	6	40	21	39
8	7	6	14	10	14
9	7	6	28	22	28
-13	8	6	58	40	41
-12	8	6	29	3	28
-11	8	6	47	35	47
-10	8	6	0	8	1
-9	8	6	95	88	14
-8	8	6	54	62	26
-7	8	6	65	53	29
-6	8	6	58	46	29
-5	8	6	98	86	11
-4	8	6	93	68	20
-3	8	6	178	170	8
-2	8	6	212	222	7
-1	8	6	187	186	7
0	8	6	62	48	16
1	8	6	63	70	16
2	8	6	37	43	37
3	8	6	25	45	24
4	8	6	205	206	8
5	8	6	127	134	9
6	8	6	27	13	27
7	8	6	10	28	10
8	8	6	34	13	33
-12	9	6	42	24	41
-11	9	6	0	10	1
-10	9	6	28	20	27
-9	9	6	5	16	4

Table 6. Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
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-2	9	6	152	135	14
-1	9	6	72	71	15
0	9	6	204	206	7
1	9	6	62	68	18
2	9	6	29	32	28
3	9	6	188	207	8
4	9	6	0	9	1
5	9	6	14	8	14
6	9	6	35	42	34
7	9	6	53	55	43
-11	10	6	90	63	32
-10	10	6	32	34	31
-9	10	6	38	33	38
-8	10	6	33	4	33
0	10	6	31	40	31
1	10	6	103	96	11
2	10	6	24	24	24
3	10	6	0	7	1
4	10	6	0	28	1
5	10	6	0	4	1
6	10	6	49	28	48
-10	11	6	57	33	57
2	11	6	48	33	48
3	11	6	51	55	43
4	11	6	21	31	21
5	11	6	60	28	59
-17	1	7	0	2	1
-16	1	7	26	39	25
-15	1	7	140	117	7
-14	1	7	36	41	36
-13	1	7	39	41	23
-12	1	7	153	150	5
-11	1	7	130	126	5
-10	1	7	18	7	17
-9	1	7	50	44	11
-8	1	7	51	55	10
-7	1	7	112	113	4
-6	1	7	51	46	8
-5	1	7	326	341	5
-4	1	7	87	98	5
-3	1	7	161	158	5
-2	1	7	11	23	10
-1	1	7	109	101	7
0	1	7	213	213	5
1	1	7	121	106	5
2	1	7	58	54	10
3	1	7	59	61	10
4	1	7	80	95	8
5	1	7	193	192	5
6	1	7	24	19	23
7	1	7	59	80	18
8	1	7	54	63	24
9	1	7	61	56	55
10	1	7	0	16	1
-17	2	7	23	40	22
-16	2	7	91	72	12
-15	2	7	0	20	1
-14	2	7	99	88	10
-13	2	7	79	66	9
-12	2	7	72	71	9
-11	2	7	71	72	8
-10	2	7	107	113	5
-9	2	7	131	126	5
-8	2	7	71	71	7
-7	2	7	78	70	7
-6	2	7	298	300	5
-5	2	7	160	161	4
-4	2	7	101	107	5
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-2	2	7	502	491	8
-1	2	7	42	58	16
0	2	7	81	74	9
1	2	7	93	73	8
2	2	7	65	61	9
3	2	7	37	33	20
4	2	7	583	580	8
5	2	7	91	86	11
6	2	7	150	147	8
7	2	7	114	132	12
8	2	7	43	19	42
9	2	7	25	42	25
10	2	7	39	27	38
11	2	7	43	39	42
-16	3	7	59	52	32
-15	3	7	30	36	29
-14	3	7	14	7	14
-13	3	7	19	8	19
-12	3	7	133	123	9
-11	3	7	204	190	6
-10	3	7	14	29	13
-9	3	7	61	62	9
-8	3	7	74	73	7
-7	3	7	134	133	4
-6	3	7	142	146	5
-5	3	7	139	120	5
-4	3	7	293	301	5
-3	3	7	40	35	15
-2	3	7	14	10	14
-1	3	7	67	63	8
0	3	7	96	89	8
1	3	7	275	247	6
2	3	7	75	68	9
3	3	7	55	52	13
4	3	7	49	52	23
5	3	7	148	143	8
6	3	7	108	116	10
7	3	7	51	77	24
8	3	7	52	50	26
9	3	7	23	19	22
10	3	7	0	38	1
11	3	7	69	67	24
-15	4	7	23	22	22
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-8	4	7	39	48	14
-7	4	7	142	191	13
-6	4	7	87	80	8
-5	4	7	34	44	34
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-3	4	7	56	48	10
-2	4	7	165	141	4
-1	4	7	345	330	5
0	4	7	154	141	7
1	4	7	0	14	1
2	4	7	172	166	8
3	4	7	23	32	23
4	4	7	100	108	10
5	4	7	144	152	8
6	4	7	0	14	1
7	4	7	25	16	25
8	4	7	94	90	13
9	4	7	37	18	37
10	4	7	29	4	29
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-14	5	7	6	5	5
-13	5	7	35	32	34
-12	5	7	0	12	1
-11	5	7	124	119	7
-10	5	7	24	28	24
-9	5	7	17	18	17
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-7	5	7	85	100	9
-6	5	7	141	150	6
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0	5	7	171	170	8
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2	5	7	37	28	36
3	5	7	61	77	17
4	5	7	52	63	19
5	5	7	152	147	13
6	5	7	20	18	19
7	5	7	23	26	22
8	5	7	108	114	12
9	5	7	70	51	20
10	5	7	28	17	27
-14	6	7	26	18	26
-13	6	7	35	44	34
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-6	6	7	84	86	8
-5	6	7	454	430	6
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-3	6	7	0	15	1
-2	6	7	29	31	28
-1	6	7	252	228	8
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1	6	7	148	154	8
2	6	7	98	111	11
3	6	7	25	22	25
4	6	7	161	171	8
5	6	7	13	12	12
6	6	7	42	59	41
7	6	7	58	57	21
8	6	7	52	45	28
9	6	7	31	1	31
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0	7	7	49	45	26
1	7	7	159	169	8
2	7	7	45	57	28
3	7	7	22	26	22
4	7	7	84	90	13
5	7	7	128	149	14
6	7	7	45	49	45
7	7	7	62	76	20
8	7	7	23	12	22
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-9	8	7	0	28	1
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-7	8	7	0	12	1
-6	8	7	50	57	29
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-4	8	7	29	23	28
-3	8	7	25	10	25
-2	8	7	177	167	8
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0	8	7	0	27	1
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2	8	7	8	9	7
3	8	7	0	0	1
4	8	7	15	29	15
5	8	7	51	55	27
6	8	7	0	8	1
7	8	7	15	27	15
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-2	9	7	42	35	41
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0	9	7	0	17	1
1	9	7	35	45	35
2	9	7	126	134	10
3	9	7	58	59	21
4	9	7	10	18	9
5	9	7	26	19	25
6	9	7	50	21	39
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-1	10	7	45	31	44
0	10	7	30	34	29
1	10	7	69	85	18
2	10	7	57	111	24
3	10	7	20	42	20
4	10	7	39	63	39
5	10	7	30	50	30
1	11	7	72	61	32
2	11	7	72	80	23
3	11	7	55	60	41
4	11	7	30	9	29
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-15	0	8	26	17	26
-14	0	8	231	260	25
-13	0	8	0	27	1
-12	0	8	158	153	7
-11	0	8	0	12	1
-10	0	8	20	4	20
-9	0	8	325	323	10
-8	0	8	266	273	6
-7	0	8	40	30	20
-6	0	8	258	259	6
-5	0	8	225	227	6
-4	0	8	92	100	8
-3	0	8	372	368	12
-2	0	8	226	231	10
-1	0	8	162	161	10
0	0	8	60	66	25
1	0	8	176	202	22
2	0	8	283	265	7
3	0	8	175	176	8
4	0	8	471	443	10
5	0	8	67	82	16
6	0	8	68	64	17
7	0	8	10	6	10
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-16	1	8	40	41	40
-15	1	8	92	94	9
-14	1	8	55	51	15
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-12	1	8	0	3	1
-11	1	8	0	10	1
-10	1	8	33	35	20
-9	1	8	65	62	8
-8	1	8	24	37	24
-7	1	8	202	197	4
-6	1	8	138	141	4
-5	1	8	138	134	4
-4	1	8	173	173	5
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-2	1	8	155	156	7
-1	1	8	272	252	7
0	1	8	129	116	6
1	1	8	120	124	6
2	1	8	161	152	5
3	1	8	308	308	5
4	1	8	258	254	6
5	1	8	172	169	5
6	1	8	153	152	8
7	1	8	22	28	22
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9	1	8	23	17	22
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-7	2	8	129	125	4
-6	2	8	84	91	6
-5	2	8	8	2	8
-4	2	8	10	8	9
-3	2	8	103	106	6
-2	2	8	48	54	13
-1	2	8	54	64	14
0	2	8	60	51	13
1	2	8	23	22	23
2	2	8	187	185	5
3	2	8	149	147	5
4	2	8	93	97	12
5	2	8	138	143	8
6	2	8	34	47	34
7	2	8	49	66	31
8	2	8	31	29	31
9	2	8	26	34	25
10	2	8	0	10	1
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-15	3	8	40	38	25
-14	3	8	31	41	31
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-12	3	8	31	36	31
-11	3	8	17	14	17
-10	3	8	0	6	1
-9	3	8	108	111	5
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-6	3	8	77	71	8
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0	3	8	82	66	11
1	3	8	41	49	41
2	3	8	49	40	19
3	3	8	180	172	8
4	3	8	202	204	8
5	3	8	251	272	8
6	3	8	68	72	19
7	3	8	54	58	28
8	3	8	41	30	40
9	3	8	45	25	44
10	3	8	102	100	16
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-13	4	8	29	32	28
-12	4	8	59	64	13
-11	4	8	28	20	27
-10	4	8	40	47	17
-9	4	8	74	79	8
-8	4	8	60	63	13
-7	4	8	112	127	7
-6	4	8	109	101	7
-5	4	8	19	3	19
-4	4	8	143	136	5
-3	4	8	179	176	5
-2	4	8	148	140	5
-1	4	8	0	15	1
0	4	8	28	26	28
1	4	8	90	105	12
2	4	8	18	13	18
3	4	8	109	108	10
4	4	8	264	277	8
5	4	8	94	98	12
6	4	8	0	30	1
7	4	8	0	11	1
8	4	8	0	13	1
9	4	8	26	15	26
10	4	8	60	27	59
-15	5	8	53	26	52
-14	5	8	27	16	27
-13	5	8	19	19	18
-12	5	8	29	31	29
-11	5	8	83	96	11
-10	5	8	39	31	33
-9	5	8	47	44	17
-8	5	8	62	58	12
-7	5	8	30	45	30
-6	5	8	545	545	9
-5	5	8	109	113	7
-4	5	8	115	120	6
-3	5	8	136	126	6
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0	5	8	152	161	8
1	5	8	71	70	15
2	5	8	116	121	10
3	5	8	34	37	33
4	5	8	52	45	22
5	5	8	45	68	34
6	5	8	0	7	1
7	5	8	65	63	21
8	5	8	11	30	11
9	5	8	60	65	59
-14	6	8	39	13	39
-13	6	8	0	14	1
-12	6	8	89	93	15
-11	6	8	0	11	1
-10	6	8	98	97	10
-9	6	8	105	106	8
-8	6	8	299	292	6
-7	6	8	244	229	6
-6	6	8	103	92	7
-5	6	8	91	97	10
-4	6	8	285	282	6
-3	6	8	150	136	6
-2	6	8	178	171	8
-1	6	8	77	76	15

Table 6. . Observed and calculated structure factors for af006

h	k	l	10Fo	10Fc	10s
0	6	8	0	26	1
1	6	8	47	42	34
2	6	8	17	27	17
3	6	8	27	26	27
4	6	8	5	12	4
5	6	8	0	8	1
6	6	8	0	5	1
7	6	8	0	28	1
8	6	8	36	36	36
-14	7	8	0	4	1
-13	7	8	25	1	24
-12	7	8	31	39	30
-11	7	8	0	7	1
-10	7	8	18	11	18
-9	7	8	94	87	10
-8	7	8	0	6	1
-7	7	8	114	112	8
-6	7	8	154	153	7
-5	7	8	62	51	17
-4	7	8	49	48	19
-3	7	8	120	111	10
-2	7	8	113	113	12
-1	7	8	75	69	14
0	7	8	18	11	17
1	7	8	54	43	23
2	7	8	0	3	1
3	7	8	21	24	20
4	7	8	63	78	19
5	7	8	42	42	42
6	7	8	44	34	44
7	7	8	23	26	22
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-12	8	8	36	40	35
-11	8	8	43	36	42
-10	8	8	40	41	40
-9	8	8	53	59	53
-8	8	8	141	153	12
-7	8	8	41	36	41
-6	8	8	67	66	20
-5	8	8	94	80	23
-4	8	8	58	71	26
-3	8	8	0	12	1
-2	8	8	56	78	21
-1	8	8	164	162	12
0	8	8	58	65	23
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3	8	8	0	17	1
4	8	8	35	34	35
5	8	8	0	5	1
6	8	8	46	45	46
-12	9	8	109	70	23
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-10	9	8	6	3	5
-9	9	8	0	24	1
-8	9	8	0	1	1
-4	9	8	0	9	1
-3	9	8	0	24	1
-2	9	8	43	35	43
-1	9	8	38	47	38
0	9	8	53	33	30
1	9	8	34	33	34
2	9	8	23	26	23
3	9	8	57	54	24
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5	9	8	62	63	27
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3	10	8	8	24	7
4	10	8	40	62	40
1	11	8	0	20	1
2	11	8	17	25	17
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-16	1	9	0	26	1
-15	1	9	41	31	32
-14	1	9	58	50	13
-13	1	9	5	23	4
-12	1	9	22	12	21
-11	1	9	80	79	7
-10	1	9	0	2	1
-9	1	9	17	23	17
-8	1	9	82	93	6
-7	1	9	23	28	22
-6	1	9	71	83	7
-5	1	9	86	93	6
-4	1	9	24	27	23
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-2	1	9	62	72	15
-1	1	9	0	8	1
0	1	9	26	16	26
1	1	9	34	12	34
2	1	9	119	105	7
3	1	9	227	223	6
4	1	9	51	41	18
5	1	9	26	10	26
6	1	9	20	17	19
7	1	9	59	58	26
8	1	9	57	64	57
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-11	2	9	64	70	8
-10	2	9	153	154	5
-9	2	9	16	26	16
-8	2	9	263	263	5
-7	2	9	0	18	1
-6	2	9	39	40	13
-5	2	9	230	233	5
-4	2	9	182	188	5
-3	2	9	337	340	6
-2	2	9	131	116	8
-1	2	9	109	107	8
0	2	9	22	36	21
1	2	9	89	90	8
2	2	9	154	145	6
3	2	9	157	148	6
4	2	9	83	63	14
5	2	9	102	109	12
6	2	9	63	78	22
7	2	9	42	44	41
8	2	9	25	18	25
9	2	9	0	4	1
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4	3	9	72	65	17
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4	4	9	112	129	11
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6	4	9	58	50	27
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2	5	9	77	85	16
3	5	9	135	140	9
4	5	9	53	57	24
5	5	9	67	66	18
6	5	9	30	32	29
7	5	9	50	33	50
8	5	9	0	0	1
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-9	6	9	42	35	42
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-2	6	9	95	102	13
-1	6	9	65	51	19
0	6	9	76	96	16
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2	6	9	113	112	11
3	6	9	46	61	45
4	6	9	30	3	29
5	6	9	39	19	39
6	6	9	42	39	42
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2	7	9	85	73	14
3	7	9	5	28	4
4	7	9	34	10	33
5	7	9	37	48	37
6	7	9	48	38	47
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-11	8	9	69	66	22
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-9	8	9	37	10	36
-8	8	9	0	9	1
-7	8	9	55	50	31
-6	8	9	28	6	27
-5	8	9	0	12	1
-4	8	9	75	56	20
-3	8	9	0	2	1
-2	8	9	48	33	40
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1	8	9	65	49	21
2	8	9	0	5	1
3	8	9	12	8	12
4	8	9	11	34	11
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-10	9	9	10	5	9
-9	9	9	94	84	23
-5	9	9	51	31	51
-4	9	9	50	39	49
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2	9	9	25	43	24
3	9	9	98	81	14
4	9	9	24	8	23
-10	10	9	14	0	14
-3	10	9	99	80	28
-2	10	9	37	6	36
-1	10	9	53	44	52
0	10	9	71	57	24
1	10	9	146	141	11
2	10	9	77	68	20
0	11	9	30	24	29
1	11	9	46	56	46
-17	0	10	13	8	13
-16	0	10	41	2	41
-15	0	10	81	80	15
-14	0	10	94	83	15
-13	0	10	85	84	10
-12	0	10	24	18	24
-11	0	10	113	118	7
-10	0	10	36	46	32
-9	0	10	20	30	19
-8	0	10	172	174	6
-7	0	10	33	29	32
-6	0	10	52	56	16
-5	0	10	249	259	7
-4	0	10	150	157	11
-3	0	10	132	148	13
-2	0	10	34	40	33
-1	0	10	56	47	46
0	0	10	48	58	38
1	0	10	242	255	34
2	0	10	485	444	10
3	0	10	36	22	36
4	0	10	14	45	14
5	0	10	68	52	22
-16	1	10	55	56	13
-15	1	10	54	49	13
-14	1	10	162	170	6
-13	1	10	21	37	20
-12	1	10	57	52	11
-11	1	10	47	51	12
-10	1	10	94	95	6
-9	1	10	155	156	4
-8	1	10	126	126	5
-7	1	10	74	72	7
-6	1	10	38	45	16
-5	1	10	44	52	17
-4	1	10	263	271	6
-3	1	10	55	60	19
-2	1	10	178	180	8
-1	1	10	34	34	33
0	1	10	156	143	6
1	1	10	486	434	7
2	1	10	278	254	6
3	1	10	47	40	24
4	1	10	88	99	12
5	1	10	38	43	38
6	1	10	0	20	1
7	1	10	65	62	64
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-15	2	10	25	15	25
-14	2	10	15	6	15
-13	2	10	0	16	1
-12	2	10	55	66	10
-11	2	10	73	71	8
-10	2	10	92	94	6
-9	2	10	27	25	27
-8	2	10	90	97	6
-7	2	10	33	34	23
-6	2	10	46	53	14
-5	2	10	134	139	6
-4	2	10	42	49	20
-3	2	10	28	19	27
-2	2	10	60	46	19
-1	2	10	0	0	1
0	2	10	12	30	11
1	2	10	54	41	17
2	2	10	60	59	19
3	2	10	0	10	1
4	2	10	78	83	17
5	2	10	38	50	37
6	2	10	46	51	46
7	2	10	23	12	23
8	2	10	82	54	37
-16	3	10	0	41	1
-15	3	10	56	52	14
-14	3	10	48	41	17
-13	3	10	0	10	1
-12	3	10	46	34	15
-11	3	10	77	81	8
-10	3	10	40	49	17
-9	3	10	267	257	6
-8	3	10	0	7	1
-7	3	10	88	99	8
-6	3	10	16	13	15
-5	3	10	81	91	10
-4	3	10	0	5	1
-3	3	10	56	45	14
-2	3	10	58	72	16
-1	3	10	18	15	18
0	3	10	14	0	14
1	3	10	16	17	16
2	3	10	204	189	9
3	3	10	92	90	15
4	3	10	29	17	28
5	3	10	66	75	21
6	3	10	56	39	36
7	3	10	32	29	32
8	3	10	22	26	21
-16	4	10	0	7	1
-15	4	10	53	59	19
-14	4	10	18	29	18
-13	4	10	98	85	10
-12	4	10	25	14	25
-11	4	10	32	18	32
-10	4	10	42	64	23
-9	4	10	0	18	1
-8	4	10	179	169	6
-7	4	10	190	206	6
-6	4	10	90	97	8
-5	4	10	202	206	5
-4	4	10	166	167	6
-3	4	10	156	147	6
-2	4	10	0	11	1
-1	4	10	239	220	9
0	4	10	40	4	39
1	4	10	186	176	8
2	4	10	127	114	11
3	4	10	25	11	25
4	4	10	117	111	11
5	4	10	26	12	26
6	4	10	43	39	43
7	4	10	30	39	30
8	4	10	0	36	1
-14	5	10	51	55	26
-13	5	10	31	47	30
-12	5	10	36	47	36
-11	5	10	24	3	23
-10	5	10	23	17	23
-9	5	10	51	52	18
-8	5	10	30	0	29
-7	5	10	196	196	6
-6	5	10	25	25	25
-5	5	10	77	67	12
-4	5	10	104	114	8
-3	5	10	80	72	10
-2	5	10	175	159	9
-1	5	10	73	67	17
0	5	10	111	109	12
1	5	10	136	140	10
2	5	10	105	108	13
3	5	10	71	69	19
4	5	10	0	2	1
5	5	10	29	57	29
6	5	10	35	26	35
7	5	10	37	1	37
-14	6	10	48	37	48
-13	6	10	37	36	37
-12	6	10	65	69	18
-11	6	10	16	20	16

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af005a.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	43(1)	43(1)	51(1)	5(1)	4(1)	1(1)
C(2)	43(1)	40(1)	59(1)	4(1)	2(1)	2(1)
C(3)	44(1)	42(1)	58(1)	2(1)	-2(1)	2(1)
C(4)	43(1)	44(1)	56(1)	4(1)	-4(1)	4(1)
C(5)	47(1)	41(1)	43(1)	1(1)	-4(1)	5(1)
C(6)	50(1)	49(1)	58(1)	0(1)	-3(1)	6(1)
C(7)	68(1)	58(1)	58(1)	-5(1)	1(1)	19(1)
C(8)	88(1)	47(1)	57(1)	-12(1)	-8(1)	7(1)
C(9)	67(1)	56(1)	52(1)	-4(1)	-10(1)	-7(1)
C(10)	46(1)	49(1)	44(1)	0(1)	-5(1)	1(1)
C(11)	44(1)	65(1)	52(1)	2(1)	-3(1)	-2(1)
C(12)	45(1)	77(1)	50(1)	0(1)	-4(1)	3(1)
C(13)	46(1)	79(1)	43(1)	-1(1)	-3(1)	11(1)
C(14)	48(1)	73(1)	67(1)	-4(1)	-5(1)	4(1)
C(15)	42(1)	79(1)	68(1)	-7(1)	-2(1)	4(1)
C(16)	45(1)	76(1)	46(1)	-2(1)	1(1)	9(1)
C(17)	49(1)	74(1)	67(1)	5(1)	6(1)	6(1)
C(18)	44(1)	81(1)	67(1)	5(1)	4(1)	6(1)
C(19)	46(1)	74(1)	53(1)	-3(1)	2(1)	2(1)
C(20)	43(1)	63(1)	53(1)	-3(1)	5(1)	-3(1)
C(21)	47(1)	48(1)	43(1)	-2(1)	2(1)	-1(1)
C(22)	65(1)	55(1)	53(1)	3(1)	8(1)	-10(1)
C(23)	80(1)	45(1)	55(1)	8(1)	3(1)	-1(1)
C(24)	68(1)	50(1)	57(1)	2(1)	-5(1)	15(1)
C(25)	47(1)	48(1)	56(1)	-4(1)	3(1)	3(1)
C(26)	45(1)	39(1)	47(1)	-3(1)	0(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af005a.

	x	y	z	U(eq)
H(6A)	2411	-1236	6138	63
H(7A)	2372	-3700	5450	73
H(8A)	1700	-4840	5144	77
H(9A)	1066	-3464	5436	70
H(14A)	-187	-45	6244	75
H(15A)	-726	1845	6370	75
H(17A)	180	5222	6132	76
H(18A)	722	3333	6034	77
H(22A)	-1046	8699	5900	69
H(23A)	-1659	10272	5876	72
H(24A)	-2325	9268	6355	70
H(25A)	-2376	6768	6983	60

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	148	591	37	14	4	0	323	311	4	17	9	0	0	2	1	26	2	1	15	17	14	32	4	1	48	44	1
6	0	0	2727	2747	67	16	4	0	158	168	3	19	9	0	6	12	6	27	2	1	138	136	3	33	4	1	41	31	1
8	0	0	1688	1742	32	18	4	0	87	87	6	21	9	0	114	96	7	28	2	1	26	21	26	34	4	1	28	21	2
10	0	0	162	156	5	20	4	0	183	193	4	23	9	0	95	67	15	29	2	1	118	128	4	35	4	1	86	67	7
12	0	0	55	79	6	22	4	0	134	147	4	25	9	0	67	46	52	30	2	1	17	0	16	36	4	1	0	5	1
14	0	0	625	660	12	24	4	0	145	149	5	0	10	0	44	40	44	31	2	1	54	61	8	37	4	1	0	14	1
16	0	0	42	46	10	26	4	0	179	194	4	2	10	0	23	16	23	32	2	1	10	5	10	38	4	1	39	14	38
20	0	0	776	871	15	28	4	0	63	69	12	4	10	0	49	34	32	33	2	1	76	78	6	1	5	1	88	95	3
22	0	0	602	662	12	30	4	0	78	84	10	6	10	0	64	49	20	34	2	1	6	0	6	2	5	1	41	47	8
24	0	0	466	474	10	32	4	0	67	67	11	8	10	0	30	14	29	35	2	1	38	26	20	3	5	1	361	339	10
26	0	0	998	986	19	34	4	0	81	68	10	10	10	0	0	9	1	36	2	1	0	12	1	4	5	1	184	183	2
28	0	0	681	677	13	36	4	0	67	65	16	12	10	0	15	25	14	37	2	1	49	38	14	5	5	1	101	109	3
30	0	0	290	309	7	38	4	0	0	27	1	14	10	0	35	3	34	38	2	1	18	14	17	6	5	1	125	126	3
32	0	0	118	123	8	1	5	0	155	153	3	16	10	0	22	26	22	39	2	1	62	50	13	7	5	1	50	52	6
34	0	0	51	80	33	3	5	0	387	374	14	18	10	0	54	50	53	40	2	1	22	9	22	8	5	1	55	49	6
36	0	0	402	368	13	5	5	0	222	218	4	20	10	0	19	3	18	1	3	1	21	8	20	9	5	1	0	4	1
38	0	0	99	89	15	7	5	0	75	69	6	1	11	0	34	15	34	2	3	1	55	59	4	10	5	1	19	22	18
40	0	0	41	18	41	9	5	0	100	108	4	3	11	0	66	69	21	3	3	1	631	648	5	11	5	1	216	218	2
1	1	0	64	68	2	11	5	0	190	190	3	5	11	0	116	89	13	4	3	1	66	62	4	12	5	1	101	99	3
3	1	0	309	375	16	13	5	0	34	35	25	7	11	0	0	15	1	5	3	1	489	476	4	13	5	1	83	91	5
5	1	0	59	66	5	15	5	0	269	271	4	1	1	1	452	507	4	6	3	1	39	39	6	14	5	1	56	60	6
7	1	0	81	86	5	17	5	0	45	42	13	2	1	1	147	156	1	7	3	1	228	219	2	15	5	1	372	382	3
9	1	0	206	216	3	19	5	0	0	3	1	3	1	1	2305	2140	19	8	3	1	92	92	2	16	5	1	68	71	6
11	1	0	129	134	3	21	5	0	64	75	8	4	1	1	249	271	2	9	3	1	370	358	3	17	5	1	144	143	3
13	1	0	13	15	13	23	5	0	62	67	8	5	1	1	559	603	4	10	3	1	29	25	10	18	5	1	25	30	24
15	1	0	119	119	2	25	5	0	52	54	9	6	1	1	22	24	22	11	3	1	183	187	2	19	5	1	60	58	6
17	1	0	97	97	4	27	5	0	43	53	11	7	1	1	169	187	2	12	3	1	122	123	2	20	5	1	131	138	3
19	1	0	32	28	18	29	5	0	70	81	7	8	1	1	184	188	2	13	3	1	144	145	2	21	5	1	77	89	5
21	1	0	0	4	1	31	5	0	81	71	10	9	1	1	1393	1385	11	14	3	1	63	64	5	22	5	1	0	8	1
23	1	0	27	23	26	33	5	0	0	16	1	10	1	1	285	297	3	15	3	1	524	524	5	23	5	1	85	85	3
25	1	0	36	31	27	35	5	0	52	26	21	11	1	1	559	575	5	16	3	1	82	76	4	24	5	1	21	21	20
27	1	0	10	22	10	0	6	0	295	290	6	12	1	1	209	216	2	17	3	1	17	5	16	25	5	1	58	60	6
29	1	0	13	18	13	2	6	0	157	155	4	13	1	1	425	434	4	18	3	1	33	25	12	26	5	1	17	15	16
31	1	0	27	7	27	4	6	0	120	116	4	14	1	1	35	43	8	19	3	1	111	124	3	27	5	1	43	49	9
33	1	0	27	9	26	6	6	0	170	175	4	15	1	1	647	647	5	20	3	1	81	74	5	28	5	1	19	3	18
35	1	0	35	31	34	8	6	0	45	42	13	16	1	1	118	109	3	21	3	1	209	214	3	29	5	1	49	57	9
37	1	0	16	29	15	10	6	0	269	272	4	17	1	1	383	406	4	22	3	1	13	19	13	30	5	1	34	38	15
39	1	0	13	9	13	12	6	0	604	613	7	18	1	1	82	80	3	23	3	1	140	143	3	31	5	1	73	69	6
41	1	0	0	19	1	14	6	0	380	376	5	19	1	1	231	236	3	24	3	1	0	18	1	32	5	1	25	10	25
0	2	0	177	191	4	16	6	0	118	126	5	20	1	1	59	50	5	25	3	1	24	25	23	33	5	1	62	67	7
2	2	0	688	769	16	18	6	0	199	196	4	21	1	1	162	168	2	26	3	1	29	28	28	34	5	1	13	11	13
4	2	0	289	285	4	20	6	0	135	137	5	22	1	1	13	23	13	27	3	1	86	90	5	35	5	1	41	28	13
6	2	0	47	5	6	22	6	0	0	0	1	23	1	1	69	73	5	28	3	1	0	9	1	36	5	1	0	7	1
8	2	0	639	650	7	24	6	0	62	63	8	24	1	1	91	89	4	29	3	1	27	20	26	37	5	1	22	9	22
10	2	0	415	425	5	26	6	0	54	56	9	25	1	1	116	113	4	30	3	1	0	11	1	0	6	1	218	211	4
12	2	0	2080	1917	25	28	6	0	128	127	5	26	1	1	52	24	9	31	3	1	23	34	23	1	6	1	24	25	23
14	2	0	380	360	5	30	6	0	60	55	10	27	1	1	366	363	4	32	3	1	37	23	15	2	6	1	149	142	3
16	2	0	263	262	4	32	6	0	56	37	11	28	1	1	36	5	16	33	3	1	62	67	9	3	6	1	37	40	11
18	2	0	361	356	5	34	6	0	29	18	28	29	1	1	100	92	4	34	3	1	18	2	17	4	6	1	53	52	7
20	2	0	134	130	4	1	7	0	60	58	10	30	1	1	24	21	23	35	3	1	15	10	14	5	6	1	230	235	3
22	2	0	271	268	4	3	7	0	219	223	4	31	1	1	80	77	6	36	3	1	0	6	1	6	6	1	79	76	5
24	2	0	475	473	7	5	7	0	159	163	5	32	1	1	13	2	13	37	3	1	63	44	11	7	6	1	107	109	3
26	2	0	76	70	8	7	7	0	38	45	21	33	1	1	16	16	15	38	3	1	0	0	1	8	6	1	0	14	1
28	2	0	104	83	6	9	7	0	616	630	10	34	1	1	10	7	10	39	3	1	41	6	40	9	6	1	0	2	1
30	2	0	121	123	6	11	7	0	510	511	7	35	1	1	35	30	19	40	3	1	0	13	1	10	6	1	25	31	25
32	2	0	81	83	10	13	7	0	69	69	9	36	1	1	59	42	10	0	4	1	399	384	5	11	6	1	185	187	3
34	2	0	123	104	6	15	7	0	388	377	6	37	1	1	150	143	6	1	4	1	33	11	9	12	6	1	311	309	4
36	2	0	40	31	40	17	7	0	441	450	7	38	1	1	49	43	17	2	4	1	203	193	2	13	6	1	100	103	5
38	2	0	183	163	10	19	7	0	165	163	5	39	1	1	23	5	23	3	4	1	93	95	3	14	6	1	205	200	3
40	2	0	80	77	17	21	7	0	33	35	32	40	1	1	17	21	16	4	4	1	24	23	23	15	6	1	6	4	6
1	3	0	63	68	5	23	7	0	46	61	16	41	1	1	12	8	11	5	4	1	205	197	2	16	6	1	0	4	1
3	3	0	297	308	4	25	7	0	59	70	9	0	2	1	207	220	3	6	4	1	247	247	2	17	6	1	243	252	3
5	3	0	37	32	10	27	7	0	69	71	8	1	2	1	366	387	3	7	4	1	386	385	3	18	6	1	102	102	4
7	3	0	15	0	14	29	7	0	42	38	15	2	2	1	220	239	3	8	4	1	51	46	5	19	6	1	242	251	3
9	3	0	234	229	3	0	8	0	277	274	8	3	2	1	316	321	3	9	4	1	28	20	11	20	6	1	39	34	10
11	3	0	138	138	3	2	8	0	127	119	6	4	2	1	73	75	3	10	4	1	24	20	15	21	6	1	27	14	20
13	3	0	17	10	16	4	8	0	180	172	5	5	2	1	196	209	2	11	4	1	390	404	3	22	6	1	17	8	16
15	3	0	251	248	4	6	8	0	0	19	1	6	2	1	69	67	3	12	4	1	51	48	5	23	6	1	88	99	4
17	3	0	87	79	5	8	8	0	18	27	17	7	2	1	579	587	4	13	4	1	306	304	3	24	6	1	34	38	11
19	3	0	18	26	17	10	8	0	60	65	11	8	2	1	6	6	6	14	4	1	64	60	5	25	6	1	0	20	1
21	3	0	35	36	19	12	8	0	32	33	31	9	2	1	6	6	6	15	4	1	169	176	2	26	6	1	39	34	10
23	3	0	57	60	10	14	8	0	16	14	15	10	2	1	16	15	15	16	4	1	0	13	1	27	6	1	46	50	8
25	3	0	18	5	17	16	8	0	0	12	1	11	2	1	83	96	3	17	4	1	98	99	3	28	6	1	64	62	5
27	3	0	41	42	19	18	8	0	38	19	25	12	2	1	225	228	2	18	4	1	24	25	23	29	6	1	17	15	16
29	3	0	53	47	13	20	8	0	34	17	33	13	2	1	55	71	4	19	4	1	59	59	6	30	6	1	0	9	1
31	3	0	13	2	13	22	8	0	194	177	6	14	2	1	93	93	3	20	4	1	57	62	7	31	6	1	0	2	1
33	3	0	29	14	28	24	8	0	41	35	21	15	2	1	193	192	2	21	4	1	56	55	7	32	6	1	27	18	18
35	3	0	29	15	28	26	8	0	143	125	7	16	2	1	45	46	6	22	4	1	0	17	1	33	6	1	10	0	10
37	3	0	30	24	30	28	8	0	30	28	30	17	2	1	86	73	3	23	4	1	158	164	3	34	6	1	15	3	14
39	3	0	0	8	1	1	9	0	100	96	7	18	2	1	0	11	1	24	4	1	60	63	6	1	7	1	41	39	12
0	4	0	1305	1195	20	3	9	0	158	155	6	19	2	1	23	32	23	25	4	1	81	76	4	2	7	1	28	30	28
2	4	0	345	339	5	5	9	0	27	33	27	20	2	1	8	13	8	26	4	1	58	58	6	3	7	1	230	231	3
4	4	0	36	34	13	7	9	0	38	40	29	21	2	1	152	147	2	27	4	1	29	18	29	4	7	1	91	96	5
6	4	0	915	904	11	9	9	0	22	12	21	22	2	1	33	25	13	28	4	1	27	34	27	5	7	1	159	165	3
8	4	0	297	300	4	11	9	0	30	25	29	23	2	1	417	423	4	29	4	1	73	62	7	6	7	1	53	56	8
10	4	0	216	231	3	13	9	0	16	27	15	24	2	1	78	69	6	30	4	1	30	25	29	7	7	1	146	140	3
12	4	0	109	101	3	15	9	0	52	38	16	25	2	1	249	264	3	31	4	1	35	29	18	8	7	1	249	242	3

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
9	7	1	450	454	4
10	7	1	48	45	10
11	7	1	324	322	3
12	7	1	0	1	1
13	7	1	51	49	8
14	7	1	0	11	1
15	7	1	223	224	3
16	7	1	89	88	5
17	7	1	268	267	3
18	7	1	160	152	3
19	7	1	42	38	11
20	7	1	77	78	6
21	7	1	70	74	6
22	7	1	21	23	20
23	7	1	53	51	8
24	7	1	21	13	20
25	7	1	53	55	7
26	7	1	0	5	1
27	7	1	42	38	10
28	7	1	20	19	19
29	7	1	85	84	4
30	7	1	0	6	1
31	7	1	109	91	12
32	7	1	56	43	56
0	8	1	323	318	6
1	8	1	0	11	1
2	8	1	179	175	3
3	8	1	79	64	6
4	8	1	45	46	9
5	8	1	176	175	3
6	8	1	90	94	5
7	8	1	99	103	4
8	8	1	120	116	4
9	8	1	37	33	13
10	8	1	99	97	4
11	8	1	48	50	9
12	8	1	23	21	23
13	8	1	42	46	11
14	8	1	35	33	15
15	8	1	0	6	1
16	8	1	0	10	1
17	8	1	0	2	1
18	8	1	50	43	10
19	8	1	27	5	26
20	8	1	82	69	6
21	8	1	64	61	8
22	8	1	115	101	4
23	8	1	21	15	20
24	8	1	46	37	11
25	8	1	0	6	1
26	8	1	145	130	4
27	8	1	34	11	34
28	8	1	110	92	8
29	8	1	39	37	38
1	9	1	48	51	12
2	9	1	46	51	12
3	9	1	58	54	9
4	9	1	13	9	13
5	9	1	61	53	8
6	9	1	57	57	11
7	9	1	19	17	18
8	9	1	68	71	7
9	9	1	29	34	28
10	9	1	42	35	13
11	9	1	0	4	1
12	9	1	22	28	22
13	9	1	0	10	1
14	9	1	33	31	20
15	9	1	48	39	11
16	9	1	59	51	9
17	9	1	0	3	1
18	9	1	35	34	19
19	9	1	18	20	17
20	9	1	59	45	9
21	9	1	52	43	11
22	9	1	22	10	22
23	9	1	30	3	29
24	9	1	53	28	52
25	9	1	47	21	47
0	10	1	43	46	29
1	10	1	19	27	18
2	10	1	23	25	23
3	10	1	50	36	13
4	10	1	34	19	33
5	10	1	47	35	18
6	10	1	50	48	11
7	10	1	0	1	1
8	10	1	22	27	22
9	10	1	17	20	16
10	10	1	0	9	1
11	10	1	0	16	1
12	10	1	33	28	32
13	10	1	27	16	27
14	10	1	0	5	1
15	10	1	16	15	15
16	10	1	20	13	19
17	10	1	19	14	18
18	10	1	76	64	23
19	10	1	38	7	37
1	11	1	70	39	12
2	11	1	52	33	22
3	11	1	6	13	6
4	11	1	20	3	19
5	11	1	53	43	15
6	11	1	43	24	27
7	11	1	10	4	10
8	11	1	58	54	25
9	11	1	55	12	54
10	11	1	43	5	43
1	0	2	182	182	3
2	0	2	676	658	10
3	0	2	297	303	5
4	0	2	102	95	2
5	0	2	32	30	10
6	0	2	681	697	8
7	0	2	344	360	4
8	0	2	1030	1061	14
9	0	2	247	268	3
10	0	2	256	267	4
11	0	2	121	120	3
12	0	2	593	616	8
13	0	2	325	313	4
14	0	2	676	688	9
15	0	2	216	213	4
16	0	2	271	268	4
17	0	2	35	37	15
18	0	2	173	169	3
19	0	2	133	130	3
20	0	2	303	297	5
21	0	2	222	220	4
22	0	2	187	178	4
23	0	2	216	218	5
24	0	2	45	33	16
25	0	2	66	60	10
26	0	2	140	137	5
27	0	2	166	159	5
28	0	2	173	171	5
29	0	2	190	189	5
30	0	2	115	112	6
31	0	2	88	82	8
32	0	2	89	87	8
33	0	2	47	35	16
34	0	2	193	190	7
35	0	2	59	52	15
36	0	2	106	100	9
37	0	2	0	7	1
38	0	2	0	24	1
39	0	2	22	5	22
40	0	2	102	98	18
1	1	2	412	414	4
2	1	2	1461	1513	32
3	1	2	336	360	4
4	1	2	585	590	5
5	1	2	70	65	3
6	1	2	156	153	2
7	1	2	57	53	3
8	1	2	414	425	3
9	1	2	233	234	2
10	1	2	981	955	8
11	1	2	232	239	2
12	1	2	42	42	5
13	1	2	13	11	13
14	1	2	96	90	2
15	1	2	125	121	2
16	1	2	390	380	3
17	1	2	134	129	2
18	1	2	92	93	3
19	1	2	102	101	3
20	1	2	353	358	3
21	1	2	30	24	14
22	1	2	224	232	3
23	1	2	100	97	4
24	1	2	79	77	5
25	1	2	154	143	4
26	1	2	528	553	6
27	1	2	48	20	21
28	1	2	406	413	5
29	1	2	116	98	4
30	1	2	56	54	9
31	1	2	0	4	1
32	1	2	36	51	18
33	1	2	41	30	15
34	1	2	149	150	5
35	1	2	24	13	23
36	1	2	128	103	6
37	1	2	61	50	14
38	1	2	77	73	11
39	1	2	0	11	1
40	1	2	20	20	19
0	2	2	141	140	3
1	2	2	57	60	4
2	2	2	21	24	20
3	2	2	136	137	2
4	2	2	155	149	2
5	2	2	6	6	6
6	2	2	33	28	8
7	2	2	167	167	2
8	2	2	171	169	2
9	2	2	12	0	11
10	2	2	91	84	2
11	2	2	130	136	2
12	2	2	187	186	2
13	2	2	76	73	3
14	2	2	85	81	2
15	2	2	60	54	4
16	2	2	15	18	14
17	2	2	112	111	3
18	2	2	55	53	5
19	2	2	49	45	6
20	2	2	39	41	9
21	2	2	16	18	15
22	2	2	66	65	5
23	2	2	67	68	5
24	2	2	17	18	16
25	2	2	64	70	7
26	2	2	25	27	24
27	2	2	6	18	6
28	2	2	0	11	1
29	2	2	0	7	1
30	2	2	27	18	26
31	2	2	21	17	20
32	2	2	13	5	13
33	2	2	0	2	1
34	2	2	22	17	22
35	2	2	17	19	16
36	2	2	26	18	26
37	2	2	46	37	21
38	2	2	31	9	31
39	2	2	36	3	36
40	2	2	42	1	42
1	3	2	154	155	2
2	3	2	384	401	4
3	3	2	71	80	3
4	3	2	733	693	5
5	3	2	13	4	13
6	3	2	162	161	2
7	3	2	151	149	2
8	3	2	444	423	3
9	3	2	119	115	2
10	3	2	559	532	4
11	3	2	114	106	2
12	3	2	239	247	2
13	3	2	0	0	1
14	3	2	271	281	2
15	3	2	0	14	1
16	3	2	28	35	16
17	3	2	46	51	8
18	3	2	51	54	7
19	3	2	46	48	8
20	3	2	194	206	3
21	3	2	10	9	10
22	3	2	215	235	3
23	3	2	0	18	1
24	3	2	0	10	1
25	3	2	0	0	1
26	3	2	98	106	4
27	3	2	0	3	1
28	3	2	69	72	6
29	3	2	16	7	15
30	3	2	35	32	16
31	3	2	8	4	8
32	3	2	69	80	7
33	3	2	0	3	1
34	3	2	72	71	8
35	3	2	12	6	11
36	3	2	0	25	1
37	3	2	49	35	19
38	3	2	156	126	6
39	3	2	40	0	39
0	4	2	178	192	3
1	4	2	55	53	5
2	4	2	76	88	3
3	4	2	100	100	3
4	4	2	45	30	6
5	4	2	91	89	3
6	4	2	45	50	6
7	4	2	105	106	3
8	4	2	6	12	6
9	4	2	12	16	11
10	4	2	20	15	19
11	4	2	131	134	2
12	4	2	17	21	16
13	4	2	121	118	2
14	4	2	30	41	14
15	4	2	52	50	7
16	4	2	21	32	20
17	4	2	0	14	1
18	4	2	17	19	16
19	4	2	33	40	13
20	4	2	0	9	1
21	4	2	53	61	7
22	4	2	25	29	25
23	4	2	0	10	1
24	4	2	25	28	25
25	4	2	33	21	13
26	4	2	19	20	18
27	4	2	34	41	15
28	4	2	30	20	30
29	4	2	81	81	7
30	4	2	10	5	10
31	4	2	23	20	23
32	4	2	22	9	21
33	4	2	8	16	8
34	4	2	24	0	23
35	4	2	23	15	23
36	4	2	0	6	1
37	4	2	20	19	19
38	4	2	46	14	46
1	5	2	61	53	5
2	5	2	127	117	2
3	5	2	18	11	17
4	5	2	209	208	3
5	5	2	83	80	4
6	5	2	94	93	3
7	5	2	69	66	5
8	5	2	194	194	2
9	5	2	13	17	13
10	5	2	77	72	5
11	5	2	40	38	9
12	5	2	169	167	3
13	5	2	39	43	11
14	5	2	65	63	6
15	5	2	18	25	17
16	5	2	359	375	3
17	5	2	29	25	22
18	5	2	210	223	3
19	5	2	21	31	20
20	5	2	383	405	4
21	5	2	53	54	7
22	5	2	150	156	3
23	5	2	39	40	9
24	5	2	21	31	20
25	5	2	24	20	23
26	5	2	26	43	26
27	5	2	13	5	13
28	5	2	27	3	27
29	5	2	0	6	1
30	5	2	35	29	16
31	5	2	8	9	8
32	5	2	12	22	11
33	5	2	37	15	16
34	5	2	42	38	14
35	5	2	0	1	1
36	5	2	20	15	19
0	6	2	6	23	6
1	6	2	22	23	22
2	6	2	8	20	8
3	6	2	73	69	5
4	6	2	29	42	14
5	6	2	199	199	3
6	6	2	140	149	3
7	6	2	20	18	19
8	6	2	111	112	3
9	6	2	12	5	11
10	6	2	41	37	10
11	6	2	119	119	4
12	6	2	195	203	3
13	6	2	47	46	9
14	6	2	176	179	3
15	6	2	161	160	3
16	6	2	88	89	5
17	6	2	232	233	3
18	6	2	63	67	7
19	6	2	123	129	3
20	6	2	93	97	4
21	6	2	86	85	4
22	6	2	56	54	6
23	6	2	85	92	4
24	6	2	20	21	19
25	6	2	15	7	14
26	6	2	6	6	6
27	6	2	13	17	13
28	6	2	25	19	24
29	6	2	0	0	1
30	6	2	6	12	6
31	6	2	19	12	18
32	6	2	0	19	1
33	6	2	0	14	1
34	6	2	22	29	22
1	7	2	104	105	4
2	7	2	24	11	23
3	7	2	129	132	3
4	7	2	277	295	4
5	7	2	68	69	6
6	7	2	63	58	7
7	7	2	103	100	4
8	7	2	350	347	3
9	7	2	119	125	4
10	7	2	246	245	3
11	7	2	0	18	1
12	7	2	209	207	3
13	7	2	43	40	11
14	7	2	28	30	28
15	7	2	0	7	1
16	7	2	29	35	23
17	7	2	131	129	4
18	7	2	47	41	10
19	7	2	143	144	3
20	7	2	22	18	21
21	7	2	0	5	1
22	7	2	39	53	11
23	7	2	102	99	4
24	7	2	102	102	4
25	7	2	87	87	4
26	7	2	39	34	10
27	7	2	37	37	12
28	7	2	72	62	6
29	7	2	34	36	15
30	7	2	27	15	26
31	7	2	38	34	38
32	7	2	152	130	13
0	8	2	8	12	8
1	8	2	174	166	3
2	8	2	0	12	1
3	8	2	64	67	7
4	8	2	21	27	20
5	8	2	94	91	5
6	8	2	81	69	5
7	8	2	217	222	3
8	8	2	51	44	9
9	8	2	186	180	3
10	8	2	0	19	1
11	8	2	67	67	7
12	8	2	100	94	4
13	8	2	82	83	6
14	8	2	106	97	4
15	8	2	19	32	18
16	8	2	45	40	10
17	8	2	34	39	15
18	8	2	21	14	20
19	8	2	58	62	7
20	8	2	33	44	19
21	8	2	0	6	1
22	8	2	44	39	12
23	8	2	0	5	1

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
24	8	2	24	21	23
25	8	2	41	32	14
26	8	2	12	1	11
27	8	2	91	81	7
28	8	2	26	8	26
1	9	2	64	54	9
2	9	2	58	60	8
3	9	2	75	71	6
4	9	2	34	33	17
5	9	2	13	4	13
6	9	2	17	19	16
7	9	2	59	52	9
8	9	2	21	10	20
9	9	2	43	41	12
10	9	2	47	48	11
11	9	2	8	22	8
12	9	2	32	32	23
13	9	2	0	7	1
14	9	2	13	9	13
15	9	2	29	28	29
16	9	2	0	5	1
17	9	2	57	46	9
18	9	2	27	25	26
19	9	2	59	53	10
20	9	2	60	56	10
21	9	2	24	16	23
22	9	2	33	28	32
23	9	2	52	41	52
24	9	2	80	61	35
0	10	2	17	28	16
1	10	2	0	3	1
2	10	2	0	18	1
3	10	2	60	53	10
4	10	2	0	12	1
5	10	2	61	37	9
6	10	2	40	37	17
7	10	2	30	35	29
8	10	2	17	10	16
9	10	2	25	11	25
10	10	2	0	11	1
11	10	2	19	16	18
12	10	2	12	19	11
13	10	2	0	10	1
14	10	2	23	17	23
15	10	2	54	34	13
16	10	2	13	8	13
17	10	2	0	13	1
1	11	2	6	18	6
2	11	2	27	5	26
3	11	2	51	32	24
4	11	2	29	32	29
5	11	2	39	33	30
6	11	2	28	7	28
7	11	2	0	9	1
8	11	2	41	29	40
9	11	2	22	12	21
10	11	2	41	34	40
11	11	2	50	18	49
1	1	3	112	104	2
2	1	3	1634	1582	16
3	1	3	3342	3041	30
4	1	3	1207	1444	30
5	1	3	30	19	12
6	1	3	119	122	2
7	1	3	201	210	2
8	1	3	123	123	2
9	1	3	436	453	3
10	1	3	30	28	8
11	1	3	135	138	2
12	1	3	86	89	2
13	1	3	176	177	2
14	1	3	135	127	2
15	1	3	369	360	3
16	1	3	45	34	6
17	1	3	156	159	2
18	1	3	139	140	2
19	1	3	161	172	2
20	1	3	29	27	16
21	1	3	151	154	3
22	1	3	253	257	3
23	1	3	336	338	4
24	1	3	291	289	4
25	1	3	402	410	4
26	1	3	35	13	19
27	1	3	250	261	4
28	1	3	236	243	4
29	1	3	157	162	4
30	1	3	126	128	4
31	1	3	58	60	11
32	1	3	75	71	9
33	1	3	61	43	11
34	1	3	36	29	27
35	1	3	0	2	1
36	1	3	35	1	29
37	1	3	96	68	7
38	1	3	59	43	15
39	1	3	92	83	10
40	1	3	97	55	19
0	2	3	544	562	7
1	2	3	555	577	5
2	2	3	219	230	2
3	2	3	272	276	7
4	2	3	155	152	2
5	2	3	279	277	3
6	2	3	56	54	4
7	2	3	299	299	3
8	2	3	207	218	2
9	2	3	15	25	14
10	2	3	15	7	14
11	2	3	36	22	7
12	2	3	216	226	2
13	2	3	175	168	2
14	2	3	158	157	2
15	2	3	130	138	2
16	2	3	101	102	2
17	2	3	32	40	11
18	2	3	0	9	1
19	2	3	99	96	3
20	2	3	55	58	6
21	2	3	74	75	4
22	2	3	73	74	5
23	2	3	268	273	3
24	2	3	114	114	4
25	2	3	347	369	4
26	2	3	89	83	5
27	2	3	47	40	10
28	2	3	16	0	15
29	2	3	41	44	13
30	2	3	28	37	28
31	2	3	112	120	5
32	2	3	19	14	18
33	2	3	35	45	28
34	2	3	57	52	12
35	2	3	47	38	15
36	2	3	32	12	32
37	2	3	12	24	11
38	2	3	22	14	21
39	2	3	71	48	18
1	3	3	67	61	4
2	3	3	271	270	3
3	3	3	893	890	29
4	3	3	96	92	3
5	3	3	151	148	2
6	3	3	43	35	7
7	3	3	302	285	3
8	3	3	117	113	2
9	3	3	443	424	3
10	3	3	441	436	4
11	3	3	247	245	2
12	3	3	165	162	2
13	3	3	194	189	2
14	3	3	194	179	2
15	3	3	560	548	5
16	3	3	289	283	3
17	3	3	47	42	8
18	3	3	0	0	1
19	3	3	122	126	3
20	3	3	12	6	11
21	3	3	142	140	3
22	3	3	24	29	23
23	3	3	183	202	3
24	3	3	21	33	20
25	3	3	0	18	1
26	3	3	43	48	10
27	3	3	70	85	6
28	3	3	23	21	23
29	3	3	65	74	7
30	3	3	15	4	14
31	3	3	45	41	12
32	3	3	22	29	22
33	3	3	24	32	23
34	3	3	29	26	28
35	3	3	8	4	8
36	3	3	49	40	19
37	3	3	45	52	22
38	3	3	45	42	44
0	4	3	132	129	3
1	4	3	404	391	3
2	4	3	17	13	16
3	4	3	106	107	3
4	4	3	74	76	4
5	4	3	54	46	5
6	4	3	179	174	2
7	4	3	197	197	2
8	4	3	43	41	8
9	4	3	117	105	2
10	4	3	92	93	3
11	4	3	100	102	3
12	4	3	189	182	2
13	4	3	532	542	4
14	4	3	171	164	2
15	4	3	73	70	5
16	4	3	89	91	4
17	4	3	17	12	16
18	4	3	13	11	13
19	4	3	0	15	1
20	4	3	0	14	1
21	4	3	98	99	4
22	4	3	17	32	16
23	4	3	130	134	3
24	4	3	8	1	8
25	4	3	29	38	16
26	4	3	34	27	12
27	4	3	72	75	5
28	4	3	21	25	20
29	4	3	62	63	7
30	4	3	37	39	12
31	4	3	47	46	10
32	4	3	13	8	13
33	4	3	30	29	30
34	4	3	36	21	17
35	4	3	67	50	8
36	4	3	27	15	27
37	4	3	65	47	31
1	5	3	81	84	4
2	5	3	220	211	2
3	5	3	334	318	3
4	5	3	156	152	3
5	5	3	86	94	4
6	5	3	8	7	8
7	5	3	42	41	10
8	5	3	106	101	3
9	5	3	153	162	3
10	5	3	8	14	8
11	5	3	319	316	3
12	5	3	6	3	6
13	5	3	94	95	4
14	5	3	97	99	4
15	5	3	485	501	4
16	5	3	45	47	10
17	5	3	16	22	15
18	5	3	54	51	8
19	5	3	0	7	1
20	5	3	55	56	7
21	5	3	69	72	6
22	5	3	85	85	5
23	5	3	63	61	6
24	5	3	43	45	9
25	5	3	74	67	6
26	5	3	19	16	18
27	5	3	33	34	13
28	5	3	52	59	8
29	5	3	29	43	29
30	5	3	44	48	12
31	5	3	71	72	7
32	5	3	6	2	6
33	5	3	44	43	13
34	5	3	35	37	24
35	5	3	75	64	10
36	5	3	18	6	17
0	6	3	152	136	4
1	6	3	52	54	7
2	6	3	92	86	4
3	6	3	40	46	10
4	6	3	80	82	5
5	6	3	324	336	3
6	6	3	0	6	1
7	6	3	208	213	3
8	6	3	37	40	13
9	6	3	172	177	3
10	6	3	111	109	4
11	6	3	396	404	4
12	6	3	172	170	3
13	6	3	404	399	4
14	6	3	181	180	3
15	6	3	237	238	3
16	6	3	78	76	5
17	6	3	110	112	3
18	6	3	60	56	6
19	6	3	349	361	4
20	6	3	67	64	6
21	6	3	84	79	5
22	6	3	0	14	1
23	6	3	104	114	4
24	6	3	19	20	18
25	6	3	0	4	1
26	6	3	43	50	7
27	6	3	43	51	7
28	6	3	41	48	7
29	6	3	29	35	12
30	6	3	38	39	8
31	6	3	22	19	21
32	6	3	16	17	15
33	6	3	75	62	10
34	6	3	24	9	23
1	7	3	158	163	3
2	7	3	47	41	9
3	7	3	194	206	3
4	7	3	62	71	7
5	7	3	156	161	4
6	7	3	68	66	6
7	7	3	132	125	3
8	7	3	78	71	6
9	7	3	209	200	3
10	7	3	194	195	3
11	7	3	0	0	1
12	7	3	80	72	6
13	7	3	40	40	12
14	7	3	94	91	5
15	7	3	64	66	7
16	7	3	90	91	5
17	7	3	30	31	21
18	7	3	49	46	8
19	7	3	68	66	6
20	7	3	83	80	5
21	7	3	6	13	6
22	7	3	0	15	1
23	7	3	176	165	4
24	7	3	6	26	6
25	7	3	129	115	4
26	7	3	18	4	17
27	7	3	52	44	7
28	7	3	17	8	16
29	7	3	51	50	7
30	7	3	36	3	25
31	7	3	41	51	40
0	8	3	81	73	8
1	8	3	112	110	4
2	8	3	51	48	9
3	8	3	57	51	8
4	8	3	43	38	11
5	8	3	108	103	4
6	8	3	194	186	3
7	8	3	75	74	6
8	8	3	254	247	3
9	8	3	23	7	23
10	8	3	0	7	1
11	8	3	57	57	8
12	8	3	218	213	3
13	8	3	25	20	25
14	8	3	228	226	3
15	8	3	58	53	8
16	8	3	61	62	8
17	8	3	41	40	12
18	8	3	81	84	6
19	8	3	34	30	17
20	8	3	101	104	5
21	8	3	20	29	19
22	8	3	20	8	19
23	8	3	33	26	19
24	8	3	18	6	17
25	8	3	69	56	7
26	8	3	20	3	19
27	8	3	35	29	34
28	8	3	88	64	13
1	9	3	47	41	10
2	9	3	121	110	4
3	9	3	66	59	7
4	9	3	22	23	2
5	9	3	35	32	[illegible]
6	9	3	32	33	2
7	9	3	29	23	29
8	9	3	0	19	1
9	9	3	46	43	13
10	9	3	0	4	1
11	9	3	8	6	8
12	9	3	0	14	1
13	9	3	0	7	1
14	9	3	0	0	1
15	9	3	34	35	20
16	9	3	0	11	1
17	9	3	0	0	1
18	9	3	32	35	26
19	9	3	0	14	1
20	9	3	54	48	11
21	9	3	64	60	9
22	9	3	13	14	13
0	10	3	100	97	9
1	10	3	0	23	1
2	10	3	0	18	1
3	10	3	58	53	10
4	10	3	34	23	19
5	10	3	76	65	7
6	10	3	74	67	7
7	10	3	74	69	8
8	10	3	32	27	31
9	10	3	0	14	1
10	10	3	21	21	20
11	10	3	64	53	10
12	10	3	70	58	9
13	10	3	55	56	12
14	10	3	49	35	14
15	10	3	64	51	15
16	10	3	17	17	16
1	11	3	67	54	14
2	11	3	15	14	14
3	11	3	34	34	25
4	11	3	66	58	10
5	11	3	0	7	1
6	11	3	0	22	1
7	11	3	0	15	1
8	11	3	24	13	23
1	0	4	1988	1910	24
2	0	4	1832	1756	22
3	0	4	292	285	4
4	0	4	396	421	5
5	0	4	320	299	7
6	0	4	67	41	5
7	0	4	70	53	5
8	0	4	108	111	3
9	0	4	46	43	6
10	0	4	77	82	4
11	0	4	427	454	5
12	0	4	701	712	9
13	0	4	627	645	8
14	0	4	263	282	4
15	0	4	409	429	5
16	0	4	240	245	3
17	0	4	105	113	3
18	0	4	195	184	3
19	0	4	55	53	7
20	0	4	16	23	15
21	0	4	43	43	12
22	0	4	97	110	6
23	0	4	104	104	6
24	0	4	178	179	5
25	0	4	321	322	8
26	0	4	243	234	7
27	0	4	304	288	8
28	0	4	159	145	7
29	0	4	65	49	15
30	0	4	291	302	8
31	0	4	136	128	8
32	0	4	91	95	10
33	0	4	91	79	10
34	0	4	165	155	7
35	0	4	39	29	38
36	0	4	206	180	7
37	0	4	41	26	35
38	0	4	91	74	12
39	0	4	52	31	37
40	0	4	120	108	12
1	1	4	732	712	6
2	1	4	907	890	7
3	1	4	1684	1591	16

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
4	1	4	879	858	8
5	1	4	914	907	11
6	1	4	151	154	2
7	1	4	51	46	4
8	1	4	459	453	4
9	1	4	509	509	4
10	1	4	546	527	5
11	1	4	473	482	4
12	1	4	280	289	3
13	1	4	55	57	4
14	1	4	168	171	2
15	1	4	272	282	2
16	1	4	331	345	3
17	1	4	351	363	3
18	1	4	263	272	3
19	1	4	128	129	3
20	1	4	33	35	12
21	1	4	172	181	3
22	1	4	171	180	3
23	1	4	278	289	4
24	1	4	209	210	4
25	1	4	189	192	4
26	1	4	21	13	20
27	1	4	76	80	9
28	1	4	74	76	8
29	1	4	156	163	5
30	1	4	130	126	5
31	1	4	150	146	5
32	1	4	54	50	12
33	1	4	50	42	13
34	1	4	28	13	28
35	1	4	37	38	25
36	1	4	67	62	11
37	1	4	63	61	11
38	1	4	65	47	11
39	1	4	61	52	18
0	2	4	405	386	11
1	2	4	352	367	4
2	2	4	212	209	2
3	2	4	344	353	3
4	2	4	141	133	2
5	2	4	43	48	6
6	2	4	140	121	2
7	2	4	113	124	2
8	2	4	308	297	3
9	2	4	358	357	3
10	2	4	441	443	4
11	2	4	445	433	3
12	2	4	1029	991	8
13	2	4	686	659	5
14	2	4	49	49	6
15	2	4	279	277	2
16	2	4	197	198	2
17	2	4	72	75	5
18	2	4	183	186	3
19	2	4	151	150	3
20	2	4	91	90	4
21	2	4	13	7	13
22	2	4	196	203	3
23	2	4	163	165	3
24	2	4	349	346	4
25	2	4	148	147	4
26	2	4	155	145	4
27	2	4	0	7	1
28	2	4	10	8	10
29	2	4	49	45	13
30	2	4	80	79	8
31	2	4	21	15	20
32	2	4	89	87	7
33	2	4	0	10	1
34	2	4	86	77	8
35	2	4	83	69	9
36	2	4	60	54	12
37	2	4	45	33	20
38	2	4	114	82	10
1	3	4	242	238	3
2	3	4	506	487	4
3	3	4	259	251	2
4	3	4	271	259	3
5	3	4	267	261	3
6	3	4	34	36	12
7	3	4	91	91	4
8	3	4	30	34	13
9	3	4	282	288	3
10	3	4	51	55	5
11	3	4	202	206	2
12	3	4	0	5	1
13	3	4	13	11	13
14	3	4	62	69	5
15	3	4	233	240	3
16	3	4	6	9	6
17	3	4	79	84	4
18	3	4	43	45	8
19	3	4	13	8	13
20	3	4	0	5	1
21	3	4	104	109	3
22	3	4	61	72	6
23	3	4	47	48	8
24	3	4	95	102	4
25	3	4	36	30	13
26	3	4	46	40	9
27	3	4	27	29	27
28	3	4	73	79	6
29	3	4	44	43	17
30	3	4	48	55	15
31	3	4	42	36	18
32	3	4	16	8	15
33	3	4	22	21	22
34	3	4	17	13	16
35	3	4	44	27	18
36	3	4	13	11	13
37	3	4	38	25	37
0	4	4	647	605	9
1	4	4	259	246	3
2	4	4	74	77	5
3	4	4	80	80	4
4	4	4	141	140	2
5	4	4	218	213	2
6	4	4	492	473	4
7	4	4	153	147	2
8	4	4	401	399	3
9	4	4	58	54	6
10	4	4	223	223	2
11	4	4	25	16	25
12	4	4	167	166	3
13	4	4	181	171	3
14	4	4	51	39	8
15	4	4	33	24	16
16	4	4	179	175	3
17	4	4	44	41	10
18	4	4	180	173	3
19	4	4	25	19	25
20	4	4	79	71	5
21	4	4	95	89	4
22	4	4	79	81	5
23	4	4	16	4	15
24	4	4	104	107	4
25	4	4	68	73	6
26	4	4	106	114	4
27	4	4	41	43	11
28	4	4	37	34	14
29	4	4	19	19	18
30	4	4	36	51	16
31	4	4	43	43	13
32	4	4	0	10	1
33	4	4	57	38	12
34	4	4	48	34	20
35	4	4	28	18	28
36	4	4	54	47	23
1	5	4	61	58	6
2	5	4	182	177	3
3	5	4	239	231	3
4	5	4	111	109	3
5	5	4	176	171	2
6	5	4	50	45	8
7	5	4	28	34	19
8	5	4	135	135	3
9	5	4	187	187	3
10	5	4	302	303	3
11	5	4	109	107	4
12	5	4	137	134	3
13	5	4	51	53	8
14	5	4	225	234	3
15	5	4	20	20	19
16	5	4	199	207	3
17	5	4	130	130	4
18	5	4	37	35	14
19	5	4	30	35	20
20	5	4	62	58	7
21	5	4	57	59	7
22	5	4	67	61	6
23	5	4	8	15	8
24	5	4	69	60	6
25	5	4	26	21	26
26	5	4	18	26	17
27	5	4	37	31	12
28	5	4	33	39	15
29	5	4	40	38	12
30	5	4	32	37	18
31	5	4	57	44	9
32	5	4	37	29	20
33	5	4	43	36	16
34	5	4	44	37	43
0	6	4	140	131	4
1	6	4	177	171	3
2	6	4	90	89	4
3	6	4	44	40	10
4	6	4	37	41	10
5	6	4	0	9	1
6	6	4	341	348	3
7	6	4	38	36	13
8	6	4	186	197	3
9	6	4	72	70	6
10	6	4	281	275	3
11	6	4	42	47	12
12	6	4	563	560	5
13	6	4	228	229	3
14	6	4	412	419	4
15	6	4	60	56	8
16	6	4	16	21	15
17	6	4	47	44	8
18	6	4	186	197	3
19	6	4	0	9	1
20	6	4	20	14	19
21	6	4	34	35	12
22	6	4	105	108	4
23	6	4	37	44	10
24	6	4	44	44	7
25	6	4	21	26	20
26	6	4	60	68	5
27	6	4	61	61	5
28	6	4	18	7	17
29	6	4	25	23	16
30	6	4	80	82	5
31	6	4	18	12	17
32	6	4	107	78	8
33	6	4	21	7	20
1	7	4	28	26	28
2	7	4	177	190	3
3	7	4	0	16	1
4	7	4	155	170	4
5	7	4	52	47	9
6	7	4	90	79	5
7	7	4	10	0	10
8	7	4	41	34	13
9	7	4	231	227	3
10	7	4	119	114	4
11	7	4	97	94	5
12	7	4	18	23	17
13	7	4	15	12	14
14	7	4	56	58	8
15	7	4	120	118	4
16	7	4	72	72	6
17	7	4	127	131	3
18	7	4	16	18	15
19	7	4	8	20	8
20	7	4	0	7	1
21	7	4	17	16	16
22	7	4	101	88	4
23	7	4	18	21	17
24	7	4	109	103	4
25	7	4	29	23	16
26	7	4	34	24	11
27	7	4	29	31	14
28	7	4	34	31	11
29	7	4	21	11	20
30	7	4	55	45	15
31	7	4	0	6	1
0	8	4	152	161	5
1	8	4	81	81	5
2	8	4	56	63	8
3	8	4	47	43	12
4	8	4	162	163	4
5	8	4	142	139	3
6	8	4	49	49	10
7	8	4	223	228	3
8	8	4	15	1	14
9	8	4	78	71	6
10	8	4	8	9	8
11	8	4	143	142	4
12	8	4	87	77	6
13	8	4	212	208	3
14	8	4	98	84	5
15	8	4	156	150	4
16	8	4	18	8	17
17	8	4	0	9	1
18	8	4	62	57	8
19	8	4	124	131	4
20	8	4	26	18	26
21	8	4	82	81	6
22	8	4	136	124	4
23	8	4	10	2	10
24	8	4	40	33	14
25	8	4	0	1	1
26	8	4	82	73	8
27	8	4	40	40	39
1	9	4	81	73	6
2	9	4	89	84	6
3	9	4	139	127	4
4	9	4	69	77	7
5	9	4	42	45	13
6	9	4	56	58	10
7	9	4	49	50	12
8	9	4	17	2	16
9	9	4	42	35	14
10	9	4	37	30	17
11	9	4	45	32	12
12	9	4	0	12	1
13	9	4	0	16	1
14	9	4	43	34	13
15	9	4	42	33	13
16	9	4	32	37	23
17	9	4	13	29	13
18	9	4	0	1	1
19	9	4	42	33	15
20	9	4	36	42	21
21	9	4	54	52	11
0	10	4	0	9	1
1	10	4	8	15	8
2	10	4	35	26	35
3	10	4	0	10	1
4	10	4	34	29	23
5	10	4	0	2	1
6	10	4	57	57	10
7	10	4	22	2	21
8	10	4	62	44	10
9	10	4	0	14	1
10	10	4	0	2	1
11	10	4	12	3	11
12	10	4	78	69	8
13	10	4	22	31	22
14	10	4	52	55	20
15	10	4	0	18	1
16	10	4	55	50	40
17	10	4	29	15	29
18	10	4	40	45	40
1	11	4	27	36	27
2	11	4	51	36	32
3	11	4	0	3	1
4	11	4	0	8	1
5	11	4	22	29	22
8	11	4	20	2	19
1	1	5	499	490	4
2	1	5	441	428	4
3	1	5	802	768	7
4	1	5	851	836	8
5	1	5	175	168	3
6	1	5	332	345	4
7	1	5	113	113	3
8	1	5	280	281	3
9	1	5	178	174	2
10	1	5	105	103	2
11	1	5	333	333	3
12	1	5	103	99	2
13	1	5	110	109	2
14	1	5	42	48	5
15	1	5	126	118	3
16	1	5	22	21	22
17	1	5	62	58	5
18	1	5	50	50	7
19	1	5	12	10	11
20	1	5	72	68	5
21	1	5	56	50	8
22	1	5	138	139	5
23	1	5	31	26	31
24	1	5	265	272	5
25	1	5	128	116	5
26	1	5	99	90	7
27	1	5	147	146	
28	1	5	86	83	
29	1	5	43	41	
30	1	5	135	138	
31	1	5	37	16	
32	1	5	136	134	5
33	1	5	60	47	11
34	1	5	47	36	15
35	1	5	33	27	33
36	1	5	18	1	17
37	1	5	29	5	28
38	1	5	30	30	29
39	1	5	78	57	18
0	2	5	23	6	23
1	2	5	159	156	2
2	2	5	210	219	2
3	2	5	38	32	8
4	2	5	326	319	3
5	2	5	87	76	3
6	2	5	116	112	2
7	2	5	112	111	3
8	2	5	80	75	4
9	2	5	144	142	2
10	2	5	64	56	5
11	2	5	64	55	4
12	2	5	320	329	3
13	2	5	160	156	2
14	2	5	266	270	2
15	2	5	48	39	6
16	2	5	164	160	2
17	2	5	76	70	5
18	2	5	0	12	1
19	2	5	71	66	6
20	2	5	84	90	5
21	2	5	69	65	6
22	2	5	252	276	4
23	2	5	383	394	5
24	2	5	135	123	5
25	2	5	224	237	5
26	2	5	92	105	7
27	2	5	81	69	8
28	2	5	47	56	15
29	2	5	182	187	5
30	2	5	47	43	15
31	2	5	41	45	18
32	2	5	22	15	21
33	2	5	40	38	20
34	2	5	24	8	23
35	2	5	10	5	10
36	2	5	61	53	12
37	2	5	70	54	11
38	2	5	22	18	21
39	2	5	70	34	23
1	3	5	8	6	8
2	3	5	0	18	1
3	3	5	372	357	4
4	3	5	141	148	2
5	3	5	281	289	3
6	3	5	240	231	3
7	3	5	220	216	3
8	3	5	41	31	10
9	3	5	27	23	12
10	3	5	20	21	19
11	3	5	28	28	17
12	3	5	131	127	3
13	3	5	107	105	3
14	3	5	56	48	7
15	3	5	165	163	3
16	3	5	89	86	4
17	3	5	74	62	6
18	3	5	0	0	1
19	3	5	92	87	5
20	3	5	183	186	3
21	3	5	150	148	3
22	3	5	40	25	12
23	3	5	138	139	3
24	3	5	76	83	5
25	3	5	6	2	6
26	3	5	29	25	28
27	3	5	64	66	7
28	3	5	0	16	1
29	3	5	29	21	29
30	3	5	26	17	26
31	3	5	0	13	1
32	3	5	30	14	30
33	3	5	20	25	19
34	3	5	37	15	24

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
35	3	5	20	10	19
36	3	5	32	19	32
37	3	5	33	36	32
0	4	5	62	65	8
1	4	5	74	68	4
2	4	5	84	76	4
3	4	5	10	11	10
4	4	5	64	64	5
5	4	5	155	147	2
6	4	5	127	127	3
7	4	5	199	193	3
8	4	5	48	44	9
9	4	5	31	22	19
10	4	5	214	208	3
11	4	5	295	287	3
12	4	5	42	40	11
13	4	5	284	274	4
14	4	5	17	21	16
15	4	5	105	98	5
16	4	5	61	58	8
17	4	5	96	91	5
18	4	5	0	4	1
19	4	5	10	3	10
20	4	5	45	50	10
21	4	5	22	24	21
22	4	5	0	18	1
23	4	5	105	110	4
24	4	5	26	21	26
25	4	5	88	93	4
26	4	5	55	59	8
27	4	5	32	33	17
28	4	5	0	3	1
29	4	5	38	37	23
30	4	5	8	14	8
31	4	5	27	11	26
32	4	5	52	36	15
33	4	5	0	5	1
34	4	5	22	23	22
35	4	5	98	77	10
1	5	5	0	1	1
2	5	5	85	86	4
3	5	5	118	118	3
4	5	5	223	225	3
5	5	5	48	46	9
6	5	5	77	73	6
7	5	5	42	38	12
8	5	5	91	90	5
9	5	5	70	69	6
10	5	5	95	96	4
11	5	5	32	30	16
12	5	5	123	127	3
13	5	5	0	4	1
14	5	5	24	26	23
15	5	5	219	214	3
16	5	5	13	12	13
17	5	5	168	165	3
18	5	5	167	168	3
19	5	5	89	85	5
20	5	5	116	114	4
21	5	5	30	26	20
22	5	5	56	60	8
23	5	5	35	42	16
24	5	5	69	69	7
25	5	5	47	46	10
26	5	5	38	31	14
27	5	5	0	0	1
28	5	5	0	15	1
29	5	5	0	0	1
30	5	5	68	65	10
31	5	5	55	52	14
32	5	5	25	2	24
33	5	5	20	24	19
0	6	5	176	167	5
1	6	5	124	114	4
2	6	5	96	96	5
3	6	5	73	69	6
4	6	5	62	63	8
5	6	5	13	10	13
6	6	5	59	56	8
7	6	5	292	295	4
8	6	5	186	180	3
9	6	5	47	49	11
10	6	5	22	33	21
11	6	5	25	33	25
12	6	5	35	39	15
13	6	5	313	319	4
14	6	5	99	102	4
15	6	5	206	201	3
16	6	5	51	61	8
17	6	5	163	173	3
18	6	5	0	11	1
19	6	5	93	90	4
20	6	5	27	17	20
21	6	5	186	183	4
22	6	5	0	9	1
23	6	5	17	20	16
24	6	5	61	68	5
25	6	5	15	5	14
26	6	5	22	18	21
27	6	5	49	53	5
28	6	5	47	41	12
29	6	5	55	43	10
30	6	5	15	8	14
31	6	5	30	12	29
1	7	5	51	61	10
2	7	5	57	47	10
3	7	5	17	30	16
4	7	5	79	80	6
5	7	5	42	32	11
6	7	5	12	2	11
7	7	5	213	207	3
8	7	5	181	177	3
9	7	5	38	32	15
10	7	5	50	47	10
11	7	5	60	58	8
12	7	5	8	4	8
13	7	5	0	8	1
14	7	5	36	38	14
15	7	5	64	59	7
16	7	5	62	59	6
17	7	5	84	80	5
18	7	5	108	111	4
19	7	5	105	111	3
20	7	5	33	41	13
21	7	5	10	10	10
22	7	5	29	31	18
23	7	5	49	61	7
24	7	5	19	12	18
25	7	5	31	29	12
26	7	5	19	2	18
27	7	5	0	9	1
28	7	5	6	3	6
29	7	5	34	13	34
30	7	5	8	24	8
0	8	5	38	37	37
1	8	5	43	48	18
2	8	5	55	51	11
3	8	5	118	106	5
4	8	5	90	95	5
5	8	5	101	95	5
6	8	5	151	148	3
7	8	5	95	94	6
8	8	5	15	3	14
9	8	5	30	41	29
10	8	5	84	83	6
11	8	5	27	12	26
12	8	5	98	94	6
13	8	5	81	79	7
14	8	5	90	83	5
15	8	5	76	68	6
16	8	5	84	77	6
17	8	5	71	61	7
18	8	5	0	10	1
19	8	5	17	14	16
20	8	5	118	120	4
21	8	5	0	3	1
22	8	5	62	64	8
23	8	5	20	34	19
24	8	5	13	10	13
25	8	5	32	41	31
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1	9	5	13	7	13
2	9	5	44	42	12
3	9	5	89	76	6
4	9	5	87	93	6
5	9	5	141	134	5
6	9	5	102	99	5
7	9	5	0	12	1
8	9	5	20	24	19
9	9	5	0	2	1
10	9	5	101	98	5
11	9	5	23	27	23
12	9	5	67	65	8
13	9	5	20	22	19
14	9	5	0	5	1
15	9	5	26	25	26
16	9	5	45	42	13
17	9	5	44	32	15
18	9	5	54	50	10
19	9	5	0	7	1
20	9	5	0	13	1
21	9	5	0	16	1
22	9	5	31	35	31
23	9	5	62	54	39
0	10	5	43	35	42
1	10	5	33	37	16
2	10	5	17	6	16
3	10	5	0	14	1
4	10	5	39	47	20
5	10	5	58	56	11
6	10	5	0	9	1
7	10	5	20	24	19
8	10	5	0	6	1
9	10	5	64	55	9
10	10	5	8	1	8
11	10	5	39	39	21
12	10	5	0	4	1
13	10	5	0	1	1
14	10	5	0	16	1
15	10	5	21	27	20
1	11	5	82	47	28
2	11	5	42	2	41
3	11	5	0	19	1
4	11	5	0	10	1
0	0	6	548	556	8
1	0	6	613	608	7
2	0	6	238	239	3
3	0	6	263	272	3
4	0	6	123	123	3
5	0	6	354	366	4
6	0	6	105	98	3
7	0	6	51	51	6
8	0	6	299	293	5
9	0	6	291	280	4
10	0	6	180	175	3
11	0	6	130	129	4
12	0	6	143	137	3
13	0	6	333	312	4
14	0	6	112	99	6
15	0	6	87	82	8
16	0	6	24	17	23
17	0	6	106	95	7
18	0	6	39	33	38
19	0	6	54	57	16
20	0	6	40	41	29
21	0	6	48	37	26
22	0	6	93	90	11
23	0	6	53	58	20
24	0	6	0	19	1
25	0	6	113	102	9
26	0	6	0	5	1
27	0	6	0	15	1
28	0	6	27	38	26
29	0	6	117	104	8
30	0	6	52	51	21
31	0	6	70	74	14
32	0	6	30	25	29
33	0	6	78	67	13
34	0	6	82	76	13
35	0	6	67	55	14
36	0	6	29	23	29
37	0	6	22	22	22
38	0	6	25	5	24
1	1	6	503	482	4
2	1	6	599	554	5
3	1	6	118	118	2
4	1	6	542	509	5
5	1	6	550	534	5
6	1	6	25	21	18
7	1	6	72	71	4
8	1	6	301	300	4
9	1	6	85	84	4
10	1	6	222	220	3
11	1	6	134	133	3
12	1	6	165	164	3
13	1	6	159	165	2
14	1	6	92	82	3
15	1	6	8	7	8
16	1	6	84	71	4
17	1	6	144	150	3
18	1	6	123	119	4
19	1	6	55	44	10
20	1	6	175	173	4
21	1	6	19	12	18
22	1	6	101	94	6
23	1	6	39	40	24
24	1	6	117	115	6
25	1	6	85	80	8
26	1	6	309	315	5
27	1	6	100	101	7
28	1	6	254	264	5
29	1	6	100	92	7
30	1	6	125	127	5
31	1	6	183	183	5
32	1	6	41	30	19
33	1	6	20	15	19
34	1	6	112	101	6
35	1	6	43	40	18
36	1	6	38	24	24
37	1	6	25	20	24
38	1	6	0	5	1
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1	2	6	499	498	4
2	2	6	54	42	6
3	2	6	118	122	2
4	2	6	38	38	8
5	2	6	407	407	4
6	2	6	40	40	7
7	2	6	196	195	3
8	2	6	80	78	4
9	2	6	0	10	1
10	2	6	100	102	4
11	2	6	35	31	12
12	2	6	145	144	2
13	2	6	67	59	5
14	2	6	24	2	23
15	2	6	227	221	3
16	2	6	31	18	13
17	2	6	154	150	3
18	2	6	67	62	6
19	2	6	101	102	4
20	2	6	41	33	11
21	2	6	251	253	4
22	2	6	89	71	7
23	2	6	161	160	4
24	2	6	12	2	11
25	2	6	146	147	5
26	2	6	33	27	32
27	2	6	124	123	6
28	2	6	28	4	28
29	2	6	77	70	9
30	2	6	13	7	13
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32	2	6	32	16	31
33	2	6	39	38	22
34	2	6	29	17	28
35	2	6	47	45	15
36	2	6	24	10	23
37	2	6	50	35	49
38	2	6	24	5	23
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2	3	6	0	1	1
3	3	6	232	235	2
4	3	6	422	420	4
5	3	6	57	58	5
6	3	6	138	133	3
7	3	6	356	349	4
8	3	6	245	249	3
9	3	6	239	235	3
10	3	6	368	365	4
11	3	6	271	269	3
12	3	6	159	153	3
13	3	6	13	14	13
14	3	6	150	146	3
15	3	6	72	69	5
16	3	6	45	42	11
17	3	6	108	116	4
18	3	6	51	52	10
19	3	6	111	109	4
20	3	6	143	149	3
21	3	6	33	33	16
22	3	6	193	194	3
23	3	6	143	139	5
24	3	6	0	7	1
25	3	6	38	39	19
26	3	6	78	74	9
27	3	6	59	62	12
28	3	6	86	85	8
29	3	6	45	53	16
30	3	6	18	17	17
31	3	6	0	5	1
32	3	6	44	40	17
33	3	6	36	34	27
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35	3	6	25	23	25
36	3	6	22	14	21
37	3	6	97	93	16
0	4	6	138	131	4
1	4	6	178	182	2
2	4	6	0	10	1
3	4	6	62	55	5
4	4	6	51	47	7
5	4	6	78	90	5
6	4	6	23	14	23
7	4	6	62	62	6
8	4	6	102	100	4
9	4	6	102	96	5
10	4	6	12	14	11
11	4	6	120	109	4
12	4	6	135	132	4
13	4	6	92	90	5
14	4	6	151	151	4
15	4	6	59	59	8
16	4	6	19	5	18
17	4	6	20	27	19
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19	4	6	0	12	1
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21	4	6	62	63	7
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23	4	6	38	28	13
24	4	6	24	5	23
25	4	6	58	55	10
26	4	6	19	13	18
27	4	6	0	15	1
28	4	6	31	40	31
29	4	6	25	32	24
30	4	6	0	11	1
31	4	6	24	20	23
32	4	6	10	13	10
33	4	6	25	13	24
34	4	6	41	16	41
35	4	6	38	34	37
36	4	6	41	10	41
1	5	6	89	87	4
2	5	6	88	92	4
3	5	6	80	82	6
4	5	6	0	25	1
5	5	6	0	8	1
6	5	6	108	115	4
7	5	6	100	96	5
8	5	6	173	167	3
9	5	6	190	187	3
10	5	6	178	176	3
11	5	6	169	170	3
12	5	6	246	251	4
13	5	6	70	75	6
14	5	6	30	30	29
15	5	6	285	279	4
16	5	6	418	413	6
17	5	6	102	96	6
18	5	6	108	114	5
19	5	6	47	48	11
20	5	6	258	250	4
21	5	6	60	55	8
22	5	6	142	141	4
23	5	6	104	95	4
24	5	6	0	16	1
25	5	6	21	24	20
26	5	6	46	58	12
27	5	6	20	3	19
28	5	6	33	12	33
29	5	6	0	7	1
30	5	6	19	16	18
31	5	6	17	19	16
32	5	6	83	78	9
33	5	6	0	22	1
34	5	6	0	8	1
0	6	6	240	238	5
1	6	6	139	145	4
2	6	6	133	138	4
3	6	6	10	4	10
4	6	6	0	14	1
5	6	6	148	154	4
6	6	6	37	41	19

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
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10	6	6	60	58	8
11	6	6	0	6	1
12	6	6	98	99	5
13	6	6	79	73	6
14	6	6	0	6	1
15	6	6	54	56	8
16	6	6	18	26	17
17	6	6	164	165	4
18	6	6	15	23	14
19	6	6	80	85	5
20	6	6	52	50	7
21	6	6	68	66	5
22	6	6	70	70	5
23	6	6	38	49	8
24	6	6	22	26	21
25	6	6	57	55	9
26	6	6	95	88	5
27	6	6	84	78	7
28	6	6	99	82	6
29	6	6	24	26	23
30	6	6	0	25	1
31	6	6	8	20	8
32	6	6	18	7	17
1	7	6	85	81	6
2	7	6	24	21	23
3	7	6	0	5	1
4	7	6	109	112	5
5	7	6	0	5	1
6	7	6	207	195	4
7	7	6	142	136	4
8	7	6	225	214	4
9	7	6	98	95	6
10	7	6	34	38	22
11	7	6	113	114	5
12	7	6	0	14	1
13	7	6	0	14	1
14	7	6	0	10	1
15	7	6	41	46	11
16	7	6	43	43	11
17	7	6	86	83	5
18	7	6	30	19	17
19	7	6	131	131	4
20	7	6	33	23	15
21	7	6	17	7	16
22	7	6	30	44	15
23	7	6	41	48	8
24	7	6	65	76	6
25	7	6	21	26	20
26	7	6	0	6	1
27	7	6	42	42	8
28	7	6	48	46	24
29	7	6	28	16	28
30	7	6	0	30	1
0	8	6	91	84	11
1	8	6	38	39	15
2	8	6	49	45	13
3	8	6	0	12	1
4	8	6	18	23	17
5	8	6	149	144	4
6	8	6	8	3	8
7	8	6	66	70	9
8	8	6	53	55	11
9	8	6	75	69	8
10	8	6	30	27	29
11	8	6	37	27	20
12	8	6	0	17	1
13	8	6	47	48	13
14	8	6	16	21	15
15	8	6	0	14	1
16	8	6	32	42	31
17	8	6	22	34	21
18	8	6	50	51	11
19	8	6	15	8	14
20	8	6	0	5	1
21	8	6	67	69	8
22	8	6	49	45	9
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26	8	6	18	22	17
1	9	6	0	16	1
2	9	6	33	37	24
3	9	6	51	52	12
4	9	6	21	21	20
5	9	6	118	124	6
6	9	6	61	63	12
7	9	6	43	39	16
8	9	6	66	48	9
9	9	6	106	102	6
10	9	6	30	18	30
11	9	6	105	113	6
12	9	6	51	54	14
13	9	6	0	10	1
14	9	6	0	13	1
15	9	6	56	58	12
16	9	6	0	18	1
17	9	6	80	80	8
18	9	6	41	43	22
19	9	6	0	5	1
20	9	6	0	14	1
21	9	6	52	57	52
0	10	6	70	80	8
1	10	6	45	53	6
2	10	6	32	29	32
3	10	6	47	43	16
4	10	6	34	31	33
5	10	6	10	17	10
6	10	6	16	21	15
7	10	6	0	8	1
8	10	6	23	29	23
9	10	6	19	12	18
10	10	6	0	8	1
11	10	6	0	3	1
12	10	6	0	6	1
13	10	6	0	12	1
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3	1	7	507	484	4
4	1	7	364	361	3
5	1	7	496	489	4
6	1	7	329	315	3
7	1	7	136	131	2
8	1	7	361	355	4
9	1	7	195	185	3
10	1	7	413	415	5
11	1	7	127	117	3
12	1	7	234	230	3
13	1	7	179	163	3
14	1	7	184	181	3
15	1	7	199	187	3
16	1	7	375	368	4
17	1	7	89	82	4
18	1	7	75	76	5
19	1	7	84	84	7
20	1	7	32	23	31
21	1	7	45	47	16
22	1	7	54	50	11
23	1	7	64	60	11
24	1	7	148	146	5
25	1	7	135	136	5
26	1	7	36	33	27
27	1	7	93	85	8
28	1	7	166	166	5
29	1	7	84	79	8
30	1	7	42	34	16
31	1	7	52	48	12
32	1	7	34	29	33
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34	1	7	19	11	18
35	1	7	26	5	26
36	1	7	93	80	10
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3	2	7	225	227	2
4	2	7	515	495	4
5	2	7	168	146	3
6	2	7	685	681	7
7	2	7	211	190	3
8	2	7	315	311	5
9	2	7	32	27	19
10	2	7	55	58	7
11	2	7	32	29	18
12	2	7	177	176	3
13	2	7	107	105	4
14	2	7	271	267	3
15	2	7	23	18	23
16	2	7	68	61	6
17	2	7	49	39	10
18	2	7	192	187	4
19	2	7	50	41	16
20	2	7	300	296	5
21	2	7	72	51	10
22	2	7	312	316	5
23	2	7	194	192	4
24	2	7	197	211	4
25	2	7	166	165	5
26	2	7	379	391	6
27	2	7	95	74	8
28	2	7	173	180	5
29	2	7	20	11	19
30	2	7	36	30	25
31	2	7	45	49	17
32	2	7	111	116	8
33	2	7	49	41	14
34	2	7	48	53	16
35	2	7	32	23	32
36	2	7	62	65	25
1	3	7	65	54	5
2	3	7	229	222	2
3	3	7	418	408	4
4	3	7	116	108	3
5	3	7	173	163	3
6	3	7	85	88	5
7	3	7	216	213	4
8	3	7	82	72	6
9	3	7	30	24	29
10	3	7	222	223	4
11	3	7	181	175	3
12	3	7	179	177	3
13	3	7	135	132	3
14	3	7	59	58	8
15	3	7	91	85	4
16	3	7	169	170	3
17	3	7	147	146	4
18	3	7	83	79	6
19	3	7	109	106	5
20	3	7	65	63	8
21	3	7	103	102	6
22	3	7	48	41	14
23	3	7	142	136	5
24	3	7	97	97	7
25	3	7	0	13	1
26	3	7	8	5	8
27	3	7	44	43	19
28	3	7	15	21	14
29	3	7	17	30	16
30	3	7	36	38	35
31	3	7	0	25	1
32	3	7	37	23	26
33	3	7	25	9	24
34	3	7	35	20	35
35	3	7	43	48	43
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1	4	7	132	130	3
2	4	7	152	152	3
3	4	7	43	42	10
4	4	7	19	20	18
5	4	7	58	58	8
6	4	7	136	137	4
7	4	7	60	55	7
8	4	7	95	95	5
9	4	7	103	106	5
10	4	7	76	61	7
11	4	7	53	49	12
12	4	7	488	487	5
13	4	7	298	277	4
14	4	7	419	405	5
15	4	7	99	95	5
16	4	7	115	116	4
17	4	7	74	72	7
18	4	7	95	95	5
19	4	7	25	23	25
20	4	7	15	25	14
21	4	7	95	92	6
22	4	7	0	16	1
23	4	7	30	28	30
24	4	7	67	62	10
25	4	7	0	0	1
26	4	7	64	58	10
27	4	7	24	20	23
28	4	7	71	71	9
29	4	7	21	11	20
30	4	7	34	29	33
31	4	7	15	23	14
32	4	7	0	0	1
33	4	7	0	2	1
34	4	7	58	45	42
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3	5	7	88	87	5
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7	5	7	51	49	9
8	5	7	88	90	6
9	5	7	261	271	4
10	5	7	254	265	4
11	5	7	351	350	4
12	5	7	232	234	4
13	5	7	132	128	4
14	5	7	205	196	4
15	5	7	457	429	6
16	5	7	248	236	4
17	5	7	104	100	5
18	5	7	92	89	6
19	5	7	71	64	7
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22	5	7	26	18	26
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25	5	7	19	19	18
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28	5	7	34	24	33
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31	5	7	0	12	1
32	5	7	23	40	23
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10	6	7	146	148	4
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12	6	7	106	116	4
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15	6	7	29	27	29
16	6	7	81	92	5
17	6	7	0	13	1
18	6	7	18	31	17
19	6	7	49	66	7
20	6	7	48	63	7
21	6	7	142	148	4
22	6	7	35	31	10
23	6	7	8	11	8
24	6	7	27	29	27
25	6	7	96	80	6
26	6	7	41	37	21
27	6	7	97	84	7
28	6	7	20	7	19
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30	6	7	58	44	58
31	6	7	31	22	31
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2	7	7	116	125	5
3	7	7	43	42	15
4	7	7	73	74	8
5	7	7	57	63	9
6	7	7	40	30	14
7	7	7	109	101	5
8	7	7	65	62	8
9	7	7	106	106	5
10	7	7	130	127	5
11	7	7	53	49	11
12	7	7	21	24	20
13	7	7	24	12	23
14	7	7	29	29	29
15	7	7	26	24	26
16	7	7	35	45	15
17	7	7	15	14	14
18	7	7	29	27	22
19	7	7	6	1	6
20	7	7	24	26	23
21	7	7	21	13	20
22	7	7	70	73	5
23	7	7	48	55	7
24	7	7	75	80	5
25	7	7	22	19	22
26	7	7	10	4	10
27	7	7	55	43	19
28	7	7	49	36	36
0	8	7	45	51	17
1	8	7	77	87	6
2	8	7	0	1	1
3	8	7	63	54	10
4	8	7	58	51	13
5	8	7	38	42	18
6	8	7	38	48	19
7	8	7	23	15	23
8	8	7	109	105	5
9	8	7	17	23	16
10	8	7	6	6	6
11	8	7	18	33	17
12	8	7	71	68	8
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24	8	7	33	13	32
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2	9	7	17	15	16
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9	9	7	198	195	5
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11	9	7	186	164	5
12	9	7	34	30	33
13	9	7	34	37	28
14	9	7	16	1	15
15	9	7	141	135	5
16	9	7	24	32	23
17	9	7	125	133	7
18	9	7	22	18	22
19	9	7	55	68	55
20	9	7	17	16	16
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2	10	7	0	1	1
3	10	7	17	36	16
4	10	7	26	19	26
5	10	7	45	22	45
6	10	7	43	19	43
7	10	7	57	37	42
8	10	7	0	1	1
11	10	7	38	38	38
12	10	7	0	3	1
13	10	7	17	51	16
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2	0	8	1018	1004	11
3	0	8	488	479	5
4	0	8	271	267	3
5	0	8	594	591	8
6	0	8	90	80	6
7	0	8	184	181	4
8	0	8	72	73	10
9	0	8	0	1	1
10	0	8	144	158	8
11	0	8	333	339	7
12	0	8	57	4	15
13	0	8	436	406	10
14	0	8	234	221	6
15	0	8	260	266	6
16	0	8	40	19	39
17	0	8	0	6	1
18	0	8	83	76	11
19	0	8	205	209	5
20	0	8	12	15	11
21	0	8	34	28	34
22	0	8	72	64	13

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
23	0	8	43	29	26
24	0	8	6	19	6
25	0	8	83	69	12
26	0	8	41	40	32
27	0	8	153	156	8
28	0	8	41	38	40
29	0	8	51	43	22
30	0	8	152	148	8
31	0	8	55	50	19
32	0	8	55	58	19
33	0	8	164	153	7
34	0	8	69	60	15
35	0	8	69	57	22
1	1	8	159	149	2
2	1	8	100	86	3
3	1	8	395	381	4
4	1	8	117	114	3
5	1	8	230	223	2
6	1	8	139	131	3
7	1	8	22	23	21
8	1	8	116	112	3
9	1	8	16	4	15
10	1	8	32	29	14
11	1	8	15	8	14
12	1	8	40	34	12
13	1	8	37	32	14
14	1	8	29	27	28
15	1	8	24	15	23
16	1	8	16	7	15
17	1	8	26	19	26
18	1	8	68	58	7
19	1	8	49	51	13
20	1	8	0	4	1
21	1	8	110	107	5
22	1	8	62	54	10
23	1	8	108	93	6
24	1	8	0	8	1
25	1	8	0	2	1
26	1	8	62	61	11
27	1	8	97	100	7
28	1	8	62	56	10
29	1	8	56	51	12
30	1	8	23	24	23
31	1	8	43	32	18
32	1	8	31	28	31
33	1	8	0	5	1
34	1	8	0	3	1
35	1	8	43	13	43
0	2	8	535	530	7
1	2	8	298	294	3
2	2	8	170	164	3
3	2	8	303	300	3
4	2	8	51	41	9
5	2	8	262	255	3
6	2	8	45	16	11
7	2	8	447	439	5
8	2	8	145	134	3
9	2	8	84	80	5
10	2	8	276	269	3
11	2	8	68	62	7
12	2	8	257	263	4
13	2	8	36	34	17
14	2	8	219	210	3
15	2	8	150	147	3
16	2	8	83	83	6
17	2	8	106	104	6
18	2	8	104	105	6
19	2	8	59	53	12
20	2	8	108	100	6
21	2	8	234	228	5
22	2	8	127	120	5
23	2	8	77	66	8
24	2	8	118	108	5
25	2	8	143	138	5
26	2	8	200	198	5
27	2	8	58	42	13
28	2	8	69	59	10
29	2	8	141	144	5
30	2	8	40	37	22
31	2	8	29	22	29
32	2	8	56	39	12
33	2	8	87	93	7
34	2	8	50	51	15
35	2	8	13	6	13
1	3	8	158	154	2
2	3	8	41	30	10
3	3	8	160	154	3
4	3	8	110	104	4
5	3	8	139	134	3
6	3	8	178	174	3
7	3	8	101	99	5
8	3	8	99	95	5
9	3	8	261	262	3
10	3	8	127	123	4
11	3	8	234	235	4
12	3	8	77	71	7
13	3	8	54	45	10
14	3	8	29	23	28
15	3	8	208	202	3
16	3	8	103	94	5
17	3	8	145	145	5
18	3	8	118	117	5
19	3	8	106	109	6
20	3	8	84	79	8
21	3	8	145	141	5
22	3	8	41	29	18
23	3	8	45	41	15
24	3	8	42	49	17
25	3	8	37	45	21
26	3	8	32	22	32
27	3	8	84	93	9
28	3	8	0	5	1
29	3	8	45	54	18
30	3	8	0	3	1
31	3	8	13	5	13
32	3	8	27	27	26
33	3	8	0	8	1
34	3	8	0	0	1
0	4	8	262	259	7
1	4	8	134	130	4
2	4	8	8	0	8
3	4	8	67	71	6
4	4	8	163	168	3
5	4	8	107	107	4
6	4	8	194	193	3
7	4	8	0	15	1
8	4	8	211	202	4
9	4	8	95	88	6
10	4	8	186	179	4
11	4	8	169	168	4
12	4	8	176	168	4
13	4	8	28	10	28
14	4	8	0	1	1
15	4	8	52	40	14
16	4	8	114	109	6
17	4	8	17	22	16
18	4	8	141	132	5
19	4	8	65	58	10
20	4	8	25	24	25
21	4	8	123	114	5
22	4	8	40	22	21
23	4	8	35	25	29
24	4	8	63	49	11
25	4	8	76	74	9
26	4	8	41	37	19
27	4	8	73	65	9
28	4	8	33	28	32
29	4	8	0	13	1
30	4	8	29	3	28
31	4	8	23	9	23
32	4	8	54	33	24
33	4	8	23	29	23
1	5	8	26	30	26
2	5	8	19	8	18
3	5	8	173	175	3
4	5	8	60	56	8
5	5	8	141	146	4
6	5	8	45	48	11
7	5	8	89	93	5
8	5	8	13	13	13
9	5	8	343	351	4
10	5	8	121	119	4
11	5	8	320	318	5
12	5	8	101	99	4
13	5	8	40	39	12
14	5	8	118	107	5
15	5	8	173	158	4
16	5	8	78	68	8
17	5	8	274	263	5
18	5	8	47	21	17
19	5	8	108	93	6
20	5	8	8	35	8
21	5	8	29	36	28
22	5	8	0	17	1
23	5	8	32	30	31
24	5	8	50	52	14
25	5	8	76	55	9
26	5	8	35	29	35
27	5	8	0	1	1
28	5	8	26	20	26
29	5	8	54	35	16
30	5	8	0	22	1
31	5	8	87	58	23
0	6	8	25	17	24
1	6	8	157	172	4
2	6	8	0	6	1
3	6	8	26	23	26
4	6	8	58	57	12
5	6	8	49	53	10
6	6	8	77	83	6
7	6	8	153	151	4
8	6	8	51	46	10
9	6	8	49	54	9
10	6	8	134	138	4
11	6	8	38	46	13
12	6	8	151	155	4
13	6	8	125	126	4
14	6	8	33	44	16
15	6	8	21	21	20
16	6	8	50	56	9
17	6	8	27	32	26
18	6	8	40	53	10
19	6	8	76	83	5
20	6	8	58	67	6
21	6	8	81	91	5
22	6	8	10	17	10
23	6	8	54	48	14
24	6	8	0	16	1
25	6	8	29	26	29
26	6	8	8	24	8
27	6	8	86	72	9
28	6	8	23	31	23
29	6	8	49	29	48
1	7	8	33	46	20
2	7	8	103	111	5
3	7	8	61	72	7
4	7	8	101	102	7
5	7	8	0	9	1
6	7	8	69	66	10
7	7	8	33	27	33
8	7	8	6	6	6
9	7	8	76	65	8
10	7	8	45	48	13
11	7	8	29	34	29
12	7	8	0	8	1
13	7	8	0	6	1
14	7	8	0	7	1
15	7	8	27	13	26
16	7	8	13	17	13
17	7	8	26	27	26
18	7	8	30	38	18
19	7	8	0	1	1
20	7	8	15	15	14
21	7	8	23	33	23
22	7	8	52	58	6
23	7	8	32	40	12
24	7	8	30	40	13
25	7	8	0	8	1
26	7	8	34	12	34
0	8	8	12	19	11
1	8	8	0	8	1
2	8	8	8	9	8
3	8	8	48	45	8
4	8	8	112	105	6
5	8	8	31	14	31
6	8	8	50	48	16
7	8	8	46	49	21
8	8	8	0	8	1
9	8	8	0	12	1
10	8	8	0	27	1
11	8	8	19	16	18
12	8	8	118	113	5
13	8	8	49	33	12
14	8	8	96	93	6
15	8	8	16	9	15
16	8	8	0	17	1
17	8	8	40	42	17
18	8	8	60	57	9
19	8	8	27	30	26
20	8	8	0	13	1
21	8	8	0	4	1
22	8	8	35	62	16
1	9	8	6	19	6
2	9	8	32	35	11
3	9	8	41	52	9
4	9	8	34	47	7
5	9	8	71	69	11
6	9	8	38	50	31
7	9	8	29	26	29
8	9	8	86	81	10
9	9	8	41	39	40
10	9	8	51	51	22
11	9	8	56	47	17
12	9	8	0	21	1
13	9	8	0	4	1
14	9	8	0	9	1
15	9	8	0	27	1
16	9	8	30	36	30
17	9	8	46	48	21
18	9	8	31	64	31
0	10	8	127	103	19
1	10	8	50	26	50
2	10	8	102	86	25
3	10	8	0	9	1
4	10	8	70	33	41
1	1	9	188	182	3
2	1	9	88	79	4
3	1	9	183	182	2
4	1	9	149	148	3
5	1	9	179	173	3
6	1	9	73	70	6
7	1	9	38	40	12
8	1	9	201	197	3
9	1	9	29	15	28
10	1	9	109	112	4
11	1	9	147	143	4
12	1	9	83	89	6
13	1	9	52	50	9
14	1	9	75	73	6
15	1	9	51	51	9
16	1	9	0	7	1
17	1	9	50	50	9
18	1	9	39	35	12
19	1	9	27	25	27
20	1	9	69	57	8
21	1	9	23	40	23
22	1	9	35	13	24
23	1	9	55	53	13
24	1	9	82	86	9
25	1	9	0	14	1
26	1	9	41	31	21
27	1	9	62	52	11
28	1	9	22	20	21
29	1	9	17	21	16
30	1	9	40	36	22
31	1	9	44	38	18
32	1	9	65	66	10
33	1	9	33	2	33
0	2	9	41	32	23
1	2	9	202	201	3
2	2	9	181	171	3
3	2	9	151	137	3
4	2	9	265	256	3
5	2	9	44	36	12
6	2	9	149	148	3
7	2	9	40	3	16
8	2	9	315	316	3
9	2	9	139	139	4
10	2	9	36	35	13
11	2	9	87	75	6
12	2	9	35	29	19
13	2	9	36	35	14
14	2	9	134	137	4
15	2	9	51	40	10
16	2	9	15	19	14
17	2	9	24	26	23
18	2	9	75	66	9
19	2	9	94	85	7
20	2	9	124	127	5
21	2	9	92	80	7
22	2	9	136	131	5
23	2	9	207	202	4
24	2	9	63	56	10
25	2	9	192	184	5
26	2	9	49	41	15
27	2	9	46	36	17
28	2	9	91	75	8
29	2	9	158	154	5
30	2	9	46	34	17
31	2	9	30	32	29
32	2	9	35	24	35
33	2	9	27	3	26
1	3	9	99	85	5
2	3	9	221	221	3
3	3	9	60	45	8
4	3	9	112	108	4
5	3	9	99	95	4
6	3	9	75	72	6
7	3	9	208	210	3
8	3	9	25	12	24
9	3	9	85	88	6
10	3	9	84	83	7
11	3	9	76	69	8
12	3	9	190	190	3
13	3	9	12	11	11
14	3	9	10	20	10
15	3	9	0	11	1
16	3	9	142	142	4
17	3	9	113	113	6
18	3	9	76	77	8
19	3	9	169	165	4
20	3	9	177	176	5
21	3	9	43	39	17
22	3	9	44	50	16
23	3	9	28	1	28
24	3	9	184	191	4
25	3	9	81	83	8
26	3	9	67	58	10
27	3	9	44	43	19
28	3	9	42	42	21
29	3	9	8	17	8
30	3	9	31	41	31
31	3	9	35	18	35
32	3	9	33	39	32
0	4	9	60	59	16
1	4	9	89	88	7
2	4	9	112	114	5
3	4	9	27	29	26
4	4	9	12	9	11
5	4	9	79	69	7
6	4	9	30	27	30
7	4	9	70	69	5
8	4	9	133	136	4
9	4	9	39	29	19
10	4	9	237	233	4
11	4	9	120	112	5
12	4	9	174	171	4
13	4	9	205	195	4
14	4	9	196	190	4
15	4	9	22	8	21
16	4	9	59	50	12
17	4	9	112	97	6
18	4	9	71	72	10
19	4	9	0	6	1
20	4	9	0	7	1
21	4	9	10	3	10
22	4	9	13	23	13
23	4	9	79	84	8
24	4	9	40	30	21
25	4	9	13	9	13
26	4	9	47	52	15
27	4	9	27	16	26
28	4	9	34	24	33
29	4	9	0	17	1
30	4	9	52	43	16
31	4	9	6	8	6
1	5	9	32	36	31
2	5	9	139	142	4
3	5	9	29	17	28
4	5	9	85	86	7
5	5	9	0	4	1
6	5	9	57	55	9
7	5	9	42	46	16
8	5	9	0	10	1
9	5	9	20	21	19
10	5	9	76	85	5
11	5	9	0	0	1
12	5	9	192	202	5
13	5	9	16	3	15
14	5	9	17	28	16
15	5	9	100	86	7
16	5	9	136	123	6
17	5	9	13	14	13
18	5	9	25	34	24
19	5	9	61	54	12

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
20	5	9	120	110	6
21	5	9	16	16	15
22	5	9	45	41	18
23	5	9	45	39	19
24	5	9	80	71	9
25	5	9	53	43	14
26	5	9	27	32	26
27	5	9	22	25	21
28	5	9	46	35	19
29	5	9	19	22	18
0	6	9	32	21	32
1	6	9	21	24	20
2	6	9	41	42	14
3	6	9	82	82	7
4	6	9	30	29	29
5	6	9	68	70	8
6	6	9	19	27	18
7	6	9	0	11	1
8	6	9	10	14	10
9	6	9	109	96	6
10	6	9	0	1	1
11	6	9	86	90	5
12	6	9	87	85	5
13	6	9	27	21	27
14	6	9	50	61	9
15	6	9	20	21	19
16	6	9	58	69	7
17	6	9	64	76	6
18	6	9	34	42	13
19	6	9	124	131	5
20	6	9	52	50	6
21	6	9	60	74	5
22	6	9	8	20	8
23	6	9	0	3	1
24	6	9	46	36	20
25	6	9	0	24	1
26	6	9	10	15	10
27	6	9	25	5	24
28	6	9	0	0	1
29	6	9	0	9	1
1	7	9	15	20	14
2	7	9	53	50	10
3	7	9	34	30	17
4	7	9	43	46	11
5	7	9	8	4	8
6	7	9	34	49	13
7	7	9	114	110	7
8	7	9	38	37	38
9	7	9	19	16	18
10	7	9	42	41	24
11	7	9	0	4	1
12	7	9	37	36	26
13	7	9	0	12	1
14	7	9	58	65	7
15	7	9	0	4	1
16	7	9	31	26	19
17	7	9	18	22	17
18	7	9	21	23	20
19	7	9	57	65	8
20	7	9	8	6	8
21	7	9	13	10	13
22	7	9	21	19	20
23	7	9	0	13	1
24	7	9	13	12	13
0	8	9	0	15	1
1	8	9	37	31	14
2	8	9	49	51	10
3	8	9	84	86	7
4	8	9	23	28	23
5	8	9	0	14	1
6	8	9	20	21	19
7	8	9	87	92	10
8	8	9	53	55	19
9	8	9	69	67	13
10	8	9	0	8	1
11	8	9	37	33	36
12	8	9	44	41	23
13	8	9	64	61	14
14	8	9	45	48	19
15	8	9	73	72	12
16	8	9	28	11	28
17	8	9	63	62	10
18	8	9	26	28	26
19	8	9	36	48	23
20	8	9	26	21	26
21	8	9	29	61	29
1	9	9	41	48	10
2	9	9	34	38	12
3	9	9	38	42	10
4	9	9	24	35	15
5	9	9	25	42	11
6	9	9	18	25	12
8	9	9	10	31	10
9	9	9	52	50	19
10	9	9	50	42	36
11	9	9	0	19	1
12	9	9	52	26	52
13	9	9	0	8	1
14	9	9	19	0	18
0	10	9	85	59	28
1	10	9	120	78	18
0	0	10	149	153	7
1	0	10	77	80	9
2	0	10	162	169	4
3	0	10	154	158	4
4	0	10	81	88	8
5	0	10	0	12	1
6	0	10	96	82	6
7	0	10	96	84	6
8	0	10	22	2	21
9	0	10	192	183	5
10	0	10	74	67	12
11	0	10	100	97	9
12	0	10	81	68	11
13	0	10	200	196	7
14	0	10	91	84	9
15	0	10	32	25	32
16	0	10	26	35	26
17	0	10	108	102	7
18	0	10	54	50	15
19	0	10	85	77	9
20	0	10	25	33	25
21	0	10	39	48	25
22	0	10	25	26	25
23	0	10	0	16	1
24	0	10	0	18	1
25	0	10	0	0	1
26	0	10	0	30	1
27	0	10	16	19	15
28	0	10	30	45	30
29	0	10	45	30	28
30	0	10	6	14	6
31	0	10	21	32	20
32	0	10	0	11	1
1	1	10	424	434	5
2	1	10	75	48	8
3	1	10	266	265	4
4	1	10	270	254	4
5	1	10	235	228	3
6	1	10	52	44	10
7	1	10	50	45	10
8	1	10	94	86	5
9	1	10	42	49	13
10	1	10	91	92	6
11	1	10	0	1	1
12	1	10	51	48	10
13	1	10	63	70	8
14	1	10	47	52	9
15	1	10	24	17	23
16	1	10	55	54	8
17	1	10	27	23	26
18	1	10	47	45	9
19	1	10	49	54	11
20	1	10	98	91	6
21	1	10	30	29	29
22	1	10	51	49	13
23	1	10	41	42	19
24	1	10	131	120	6
25	1	10	151	148	5
26	1	10	170	173	5
27	1	10	82	78	9
28	1	10	102	103	7
29	1	10	126	130	6
30	1	10	80	69	10
31	1	10	138	129	6
32	1	10	18	6	17
0	2	10	60	62	17
1	2	10	66	52	9
2	2	10	15	2	14
3	2	10	96	97	6
4	2	10	94	85	6
5	2	10	0	9	1
6	2	10	6	10	6
7	2	10	110	105	4
8	2	10	100	93	5
9	2	10	136	132	4
10	2	10	0	11	1
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13	2	10	88	92	5
14	2	10	99	97	5
15	2	10	15	7	14
16	2	10	12	15	11
17	2	10	52	51	11
18	2	10	0	3	1
19	2	10	51	56	13
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22	2	10	26	10	26
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31	2	10	30	1	29
32	2	10	32	1	32
1	3	10	153	149	4
2	3	10	309	295	4
3	3	10	438	431	5
4	3	10	166	164	4
5	3	10	158	162	4
6	3	10	98	94	6
7	3	10	313	321	4
8	3	10	28	7	28
9	3	10	111	101	4
10	3	10	244	258	4
11	3	10	150	152	3
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13	3	10	90	93	5
14	3	10	79	70	6
15	3	10	66	69	8
16	3	10	95	88	5
17	3	10	27	20	27
18	3	10	139	140	5
19	3	10	236	219	5
20	3	10	84	76	8
21	3	10	22	20	22
22	3	10	32	21	32
23	3	10	242	228	5
24	3	10	175	162	5
25	3	10	125	123	6
26	3	10	20	7	19
27	3	10	33	37	33
28	3	10	45	25	18
29	3	10	81	83	9
30	3	10	107	104	8
31	3	10	0	33	1
0	4	10	37	23	36
1	4	10	71	71	9
2	4	10	83	76	7
3	4	10	10	14	10
4	4	10	15	9	14
5	4	10	99	106	6
6	4	10	22	16	22
7	4	10	183	178	4
8	4	10	143	143	4
9	4	10	83	73	6
10	4	10	98	94	5
11	4	10	195	190	4
12	4	10	68	62	9
13	4	10	224	210	4
14	4	10	96	92	5
15	4	10	195	183	5
16	4	10	22	16	21
17	4	10	39	38	24
18	4	10	28	27	28
19	4	10	146	137	5
20	4	10	51	38	15
21	4	10	124	118	5
22	4	10	25	27	25
23	4	10	27	25	27
24	4	10	15	8	14
25	4	10	12	13	11
26	4	10	0	2	1
27	4	10	37	39	23
28	4	10	0	7	1
29	4	10	54	35	16
1	5	10	64	65	8
2	5	10	42	45	15
3	5	10	144	149	4
4	5	10	81	77	7
5	5	10	53	48	11
6	5	10	43	48	13
7	5	10	15	3	14
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9	5	10	70	71	6
10	5	10	94	102	5
11	5	10	103	97	6
12	5	10	29	18	29
13	5	10	0	22	1
14	5	10	67	57	11
15	5	10	254	247	5
16	5	10	68	62	11
17	5	10	40	34	23
18	5	10	180	169	5
19	5	10	92	85	8
20	5	10	117	104	6
21	5	10	74	60	10
22	5	10	56	51	13
23	5	10	44	38	19
24	5	10	52	46	15
25	5	10	99	80	8
26	5	10	20	18	19
27	5	10	8	8	8
28	5	10	0	12	1
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6	6	10	52	54	10
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8	6	10	21	20	20
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11	6	10	21	19	20
12	6	10	71	62	11
13	6	10	113	97	7
14	6	10	0	13	1
15	6	10	32	26	31
16	6	10	28	41	19
17	6	10	63	66	6
18	6	10	21	33	20
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21	6	10	25	17	13
22	6	10	25	29	11
23	6	10	10	1	10
24	6	10	0	6	1
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26	6	10	19	2	18
1	7	10	17	27	16
2	7	10	18	28	17
3	7	10	81	80	6
4	7	10	93	107	6
5	7	10	33	35	19
6	7	10	125	131	5
7	7	10	25	32	24
8	7	10	35	42	10
9	7	10	31	45	10
10	7	10	68	67	15
11	7	10	103	97	10
12	7	10	0	5	1
13	7	10	22	22	22
14	7	10	17	3	16
15	7	10	67	56	14
16	7	10	6	20	6
17	7	10	51	16	20
18	7	10	61	50	15
19	7	10	46	54	9
20	7	10	33	29	13
21	7	10	8	4	8
22	7	10	10	3	10
23	7	10	0	4	1
0	8	10	22	20	21
1	8	10	63	70	7
2	8	10	20	14	19
3	8	10	12	17	11
4	8	10	35	36	13
5	8	10	105	105	7
6	8	10	17	3	16
7	8	10	25	21	14
8	8	10	61	76	7
9	8	10	6	7	6
10	8	10	22	27	22
11	8	10	47	43	26
12	8	10	37	32	37
13	8	10	53	37	22
14	8	10	31	32	31
15	8	10	42	37	42
17	8	10	43	17	43
18	8	10	59	46	39
19	8	10	57	48	56
1	9	10	47	56	9
2	9	10	0	15	1
3	9	10	56	57	8
4	9	10	18	30	17
5	9	10	0	8	1
6	9	10	22	49	13
7	9	10	0	2	1
8	9	10	0	16	1
9	9	10	64	47	63
10	9	10	0	7	1
1	1	11	228	233	4
2	1	11	85	84	6
3	1	11	257	266	4
4	1	11	22	15	22
5	1	11	107	95	6
6	1	11	244	244	4
7	1	11	40	27	16
8	1	11	197	196	4
9	1	11	90	78	6
10	1	11	98	91	5
11	1	11	208	201	4
12	1	11	70	71	7
13	1	11	98	90	4
14	1	11	29	41	18
15	1	11	31	26	17
16	1	11	146	151	4
17	1	11	151	152	4
18	1	11	13	1	13
19	1	11	20	25	19
20	1	11	21	25	20
21	1	11	39	24	19
22	1	11	102	96	6
23	1	11	27	24	26
24	1	11	33	29	33
25	1	11	60	61	12
26	1	11	26	6	26
27	1	11	44	45	18
28	1	11	36	30	35
29	1	11	29	29	28
30	1	11	86	77	9
31	1	11	21	22	20
0	2	11	353	345	8
1	2	11	273	279	5
2	2	11	126	115	6
3	2	11	121	126	6
4	2	11	204	200	3
5	2	11	29	23	28
6	2	11	64	55	10
7	2	11	111	119	5
8	2	11	93	85	6
9	2	11	0	22	1
10	2	11	22	24	22
11	2	11	89	87	5
12	2	11	57	67	8
13	2	11	126	127	4
14	2	11	32	35	18
15	2	11	70	66	7
16	2	11	17	5	16
17	2	11	65	63	9
18	2	11	0	4	1
19	2	11	10	0	10
20	2	11	19	11	18
21	2	11	62	54	11
22	2	11	129	125	5
23	2	11	128	126	5
24	2	11	86	88	8
25	2	11	55	40	13
26	2	11	131	126	6
27	2	11	126	118	6
28	2	11	25	9	25
29	2	11	29	3	29
30	2	11	36	31	35
1	3	11	22	30	21
2	3	11	58	34	13
3	3	11	391	400	6
4	3	11	99	80	8
5	3	11	66	68	11
6	3	11	32	22	32

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
7	3	11	38	26	16
8	3	11	232	235	4
9	3	11	212	212	4
10	3	11	179	181	4
11	3	11	63	70	8
12	3	11	37	28	15
13	3	11	89	86	5
14	3	11	94	91	5
15	3	11	141	143	4
16	3	11	110	106	5
17	3	11	119	118	6
18	3	11	208	198	5
19	3	11	45	39	16
20	3	11	61	58	11
21	3	11	13	6	13
22	3	11	83	78	8
23	3	11	175	169	5
24	3	11	65	40	10
25	3	11	59	64	11
26	3	11	25	26	24
27	3	11	27	13	27
28	3	11	22	3	21
29	3	11	55	52	17
0	4	11	98	83	9
1	4	11	43	41	18
2	4	11	57	55	11
3	4	11	63	65	10
4	4	11	10	6	10
5	4	11	88	88	7
6	4	11	208	194	5
7	4	11	109	110	6
8	4	11	206	211	5
9	4	11	33	6	22
10	4	11	115	111	6
11	4	11	164	149	5
12	4	11	369	362	5
13	4	11	172	154	4
14	4	11	370	352	5
15	4	11	209	196	5
16	4	11	73	55	10
17	4	11	6	29	6
18	4	11	105	92	7
19	4	11	10	33	10
20	4	11	58	54	12
21	4	11	98	97	7
22	4	11	98	82	7
23	4	11	49	51	16
24	4	11	66	49	11
25	4	11	35	42	35
26	4	11	41	32	21
27	4	11	42	10	21
28	4	11	70	48	30
1	5	11	44	41	15
2	5	11	96	98	6
3	5	11	21	4	20
4	5	11	113	128	5
5	5	11	48	54	13
6	5	11	39	41	18
7	5	11	16	19	15
8	5	11	0	10	1
9	5	11	34	44	20
10	5	11	59	53	9
11	5	11	21	37	20
12	5	11	119	113	6
13	5	11	70	61	12
14	5	11	125	121	6
15	5	11	136	129	6
16	5	11	97	85	8
17	5	11	45	49	18
18	5	11	65	55	11
19	5	11	0	10	1
20	5	11	30	23	29
21	5	11	0	0	1
22	5	11	82	69	9
23	5	11	8	16	8
24	5	11	77	56	10
25	5	11	30	11	29
26	5	11	20	8	19
27	5	11	0	8	1
28	5	11	55	24	55
0	6	11	125	146	7
1	6	11	43	44	13
2	6	11	37	38	17
3	6	11	32	34	31
4	6	11	0	24	1
5	6	11	33	34	22
6	6	11	50	51	12
7	6	11	90	90	7
8	6	11	23	30	23
9	6	11	44	50	11
10	6	11	47	54	8
11	6	11	22	22	21
12	6	11	27	30	14
13	6	11	21	37	20
14	6	11	43	25	26
15	6	11	57	51	17
16	6	11	47	48	21
17	6	11	0	1	1
18	6	11	33	1	33
19	6	11	23	12	23
20	6	11	0	6	1
21	6	11	63	57	15
22	6	11	56	46	22
23	6	11	15	16	14
24	6	11	20	41	7
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2	7	11	43	53	13
3	7	11	57	69	10
4	7	11	36	52	17
5	7	11	17	5	16
6	7	11	22	20	22
7	7	11	29	28	22
8	7	11	50	58	8
9	7	11	74	91	8
10	7	11	13	24	13
11	7	11	19	20	18
12	7	11	6	19	6
13	7	11	8	9	8
14	7	11	41	42	40
15	7	11	24	3	23
16	7	11	45	24	38
17	7	11	53	36	28
18	7	11	36	13	36
19	7	11	32	21	31
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21	7	11	0	15	1
0	8	11	53	66	14
1	8	11	17	38	16
2	8	11	18	0	17
3	8	11	26	26	26
4	8	11	13	17	13
5	8	11	13	9	13
6	8	11	29	38	20
7	8	11	23	15	23
8	8	11	49	75	6
9	8	11	0	3	1
10	8	11	0	7	1
11	8	11	8	7	8
13	8	11	0	18	1
1	9	11	0	1	1
2	9	11	36	10	35
3	9	11	20	15	19
4	9	11	67	48	53
5	9	11	10	24	10
0	0	12	419	456	13
1	0	12	144	158	7
2	0	12	276	293	6
3	0	12	327	333	9
4	0	12	127	142	6
5	0	12	265	276	8
6	0	12	131	134	6
7	0	12	29	32	29
8	0	12	55	54	12
9	0	12	21	34	20
10	0	12	87	93	8
11	0	12	58	52	12
12	0	12	0	16	1
13	0	12	0	13	1
14	0	12	135	132	5
15	0	12	22	10	21
16	0	12	0	12	1
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21	0	12	0	26	1
22	0	12	0	15	1
23	0	12	71	76	13
24	0	12	32	25	31
25	0	12	27	27	27
26	0	12	21	33	20
27	0	12	85	96	11
28	0	12	36	15	36
29	0	12	0	27	1
30	0	12	72	66	33
1	1	12	32	29	32
2	1	12	21	25	20
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4	1	12	41	44	15
5	1	12	28	33	28
6	1	12	105	106	6
7	1	12	31	4	31
8	1	12	116	110	5
9	1	12	15	3	14
10	1	12	54	58	9
11	1	12	36	37	15
12	1	12	21	22	20
13	1	12	0	1	1
14	1	12	41	42	11
15	1	12	0	3	1
16	1	12	26	40	26
17	1	12	10	21	10
18	1	12	64	72	8
19	1	12	23	18	23
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21	1	12	13	15	13
22	1	12	51	45	13
23	1	12	22	13	21
24	1	12	13	18	13
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0	2	12	84	81	13
1	2	12	112	114	6
2	2	12	18	29	17
3	2	12	101	103	6
4	2	12	16	21	15
5	2	12	55	47	13
6	2	12	18	9	17
7	2	12	107	108	5
8	2	12	47	41	12
9	2	12	156	158	4
10	2	12	77	79	7
11	2	12	0	23	1
12	2	12	99	101	5
13	2	12	23	10	23
14	2	12	77	93	6
15	2	12	65	64	7
16	2	12	34	33	18
17	2	12	61	56	10
18	2	12	51	51	14
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21	2	12	84	74	8
22	2	12	84	74	8
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24	2	12	34	41	34
25	2	12	12	5	11
26	2	12	69	60	11
27	2	12	94	87	8
28	2	12	16	14	15
29	2	12	71	54	30
1	3	12	38	42	22
2	3	12	154	163	5
3	3	12	64	36	12
4	3	12	269	284	5
5	3	12	99	79	10
6	3	12	232	244	6
7	3	12	47	34	14
8	3	12	35	24	24
9	3	12	47	46	12
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15	3	12	0	9	1
16	3	12	35	33	35
17	3	12	91	84	7
18	3	12	18	0	17
19	3	12	32	27	32
20	3	12	79	73	8
21	3	12	47	43	15
22	3	12	147	137	5
23	3	12	45	27	17
24	3	12	73	70	9
25	3	12	13	13	13
26	3	12	65	53	12
27	3	12	33	0	32
28	3	12	68	52	39
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1	4	12	12	1	11
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8	4	12	65	60	14
9	4	12	99	96	8
10	4	12	52	62	16
11	4	12	189	200	6
12	4	12	39	23	19
13	4	12	119	105	6
14	4	12	57	34	12
15	4	12	134	113	6
16	4	12	108	101	8
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22	4	12	62	66	12
23	4	12	48	31	18
24	4	12	45	40	20
25	4	12	32	21	31
26	4	12	0	22	1
27	4	12	17	11	16
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2	5	12	22	18	22
3	5	12	0	4	1
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6	5	12	60	59	10
7	5	12	22	5	21
8	5	12	80	84	8
9	5	12	10	3	10
10	5	12	31	26	31
11	5	12	0	11	1
12	5	12	12	21	11
13	5	12	61	52	18
14	5	12	42	38	42
15	5	12	53	42	16
16	5	12	16	31	15
17	5	12	17	11	16
18	5	12	57	50	16
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21	5	12	44	0	25
22	5	12	0	6	1
23	5	12	22	18	22
24	5	12	34	27	34
25	5	12	37	11	36
0	6	12	52	41	22
1	6	12	60	64	11
2	6	12	21	10	20
3	6	12	0	5	1
4	6	12	13	0	13
5	6	12	24	2	23
6	6	12	82	78	7
7	6	12	94	91	6
8	6	12	28	36	28
9	6	12	35	42	20
10	6	12	18	22	17
11	6	12	50	43	27
12	6	12	75	64	16
13	6	12	82	77	14
14	6	12	17	5	16
15	6	12	34	11	33
16	6	12	41	3	40
17	6	12	0	8	1
18	6	12	39	28	38
19	6	12	53	48	20
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21	6	12	54	35	41
22	6	12	30	26	29
1	7	12	0	26	1
2	7	12	29	28	28
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3	8	12	24	37	23
4	8	12	25	36	25
5	8	12	8	8	8
6	8	12	0	10	1
7	8	12	16	11	15
8	8	12	15	15	14
9	8	12	19	19	18
10	8	12	12	7	11
11	8	12	0	15	1
12	8	12	0	9	1
13	8	12	0	69	1
1	1	13	59	58	10
2	1	13	16	13	15
3	1	13	39	42	16
4	1	13	54	54	11
5	1	13	68	69	8
6	1	13	45	51	11
7	1	13	0	9	1
8	1	13	64	62	8
9	1	13	33	30	18
10	1	13	33	40	19
11	1	13	0	13	1
12	1	13	0	21	1
13	1	13	0	9	1
14	1	13	26	30	26
15	1	13	46	56	11
16	1	13	0	8	1
17	1	13	10	14	10
18	1	13	10	3	10
19	1	13	17	18	16
20	1	13	0	4	1
21	1	13	20	26	19
22	1	13	39	27	19
23	1	13	40	29	25
24	1	13	50	47	17
25	1	13	27	9	27
26	1	13	0	4	1
27	1	13	15	13	14
28	1	13	0	17	1
0	2	13	138	152	7
1	2	13	48	42	15
2	2	13	19	19	18
3	2	13	124	130	5
4	2	13	150	155	5
5	2	13	64	60	10
6	2	13	16	25	15
7	2	13	24	15	23
8	2	13	146	156	5
9	2	13	120	133	5
10	2	13	25	8	25
11	2	13	22	22	21
12	2	13	55	49	11
13	2	13	43	49	14
14	2	13	91	100	6
15	2	13	28	9	28
16	2	13	27	15	26
17	2	13	10	14	10
18	2	13	35	26	34
19	2	13	72	76	11
20	2	13	58	50	13
21	2	13	111	98	6
22	2	13	121	116	6
23	2	13	69	61	10
24	2	13	0	6	1
25	2	13	84	70	9
26	2	13	90	80	10
27	2	13	54	54	32
28	2	13	35	19	35
1	3	13	65	64	11
2	3	13	83	85	10
3	3	13	27	4	26
4	3	13	64	63	14
5	3	13	0	13	1
6	3	13	37	26	24
7	3	13	107	119	6
8	3	13	60	57	11
9	3	13	0	13	1
10	3	13	51	58	13

Table 6. Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
11	3	13	0	9	1
12	3	13	130	123	5
13	3	13	0	11	1
14	3	13	28	25	28
15	3	13	0	15	1
16	3	13	71	72	8
17	3	13	6	23	6
18	3	13	30	17	29
19	3	13	79	77	10
20	3	13	49	55	17
21	3	13	22	4	21
22	3	13	29	11	29
23	3	13	18	16	17
24	3	13	79	70	9
25	3	13	44	35	21
26	3	13	0	13	1
27	3	13	0	5	1
0	4	13	155	170	7
1	4	13	138	149	5
2	4	13	60	55	12
3	4	13	12	13	11
4	4	13	42	46	31
5	4	13	164	175	6
6	4	13	85	80	12
7	4	13	101	104	10
8	4	13	18	7	17
9	4	13	24	25	23
10	4	13	109	112	6
11	4	13	24	21	23
12	4	13	87	104	7
13	4	13	121	119	6
14	4	13	76	74	11
15	4	13	89	85	10
16	4	13	117	98	7
17	4	13	126	114	6
18	4	13	30	31	29
19	4	13	82	75	11
20	4	13	111	95	8
21	4	13	148	136	6
22	4	13	85	58	11
23	4	13	97	90	9
24	4	13	41	31	41
25	4	13	55	56	54
1	5	13	0	9	1
2	5	13	0	6	1
3	5	13	0	11	1
4	5	13	37	52	25
5	5	13	30	40	30
6	5	13	27	32	26
7	5	13	52	62	21
8	5	13	0	14	1
9	5	13	30	11	29
10	5	13	35	46	35
11	5	13	62	53	19
12	5	13	49	48	16
13	5	13	8	22	8
14	5	13	18	8	17
15	5	13	12	4	11
16	5	13	15	13	14
17	5	13	30	5	30
18	5	13	58	50	16
19	5	13	56	42	16
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0	6	13	77	75	13
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2	6	13	0	13	1
3	6	13	8	16	8
4	6	13	0	6	1
5	6	13	18	28	17
6	6	13	0	11	1
7	6	13	43	53	15
8	6	13	12	7	11
9	6	13	31	21	31
10	6	13	0	6	1
11	6	13	0	26	1
12	6	13	67	58	18
13	6	13	57	47	23
14	6	13	68	55	18
15	6	13	30	22	30
16	6	13	55	48	55
17	6	13	45	51	44
18	6	13	54	24	54
1	7	13	16	22	15
2	7	13	44	53	16
3	7	13	0	0	1
4	7	13	6	9	6
5	7	13	0	20	1
6	7	13	44	55	13
7	7	13	18	35	17
8	7	13	21	35	20
9	7	13	0	15	1
10	7	13	32	33	32
11	7	13	51	21	50
12	7	13	0	10	1
13	7	13	18	4	17
14	7	13	22	7	22
1	8	13	0	23	1
2	8	13	67	51	36
3	8	13	0	29	1
4	8	13	0	5	1
7	8	13	39	34	39
0	0	14	263	258	11
1	0	14	41	14	41
2	0	14	27	16	27
3	0	14	59	56	15
4	0	14	0	17	1
5	0	14	61	70	13
6	0	14	149	164	7
7	0	14	39	6	38
8	0	14	85	89	8
9	0	14	32	47	32
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12	0	14	72	83	9
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18	0	14	70	79	9
19	0	14	28	34	28
20	0	14	73	77	13
21	0	14	25	0	24
22	0	14	27	21	27
23	0	14	0	7	1
24	0	14	73	59	17
25	0	14	0	29	1
26	0	14	47	41	46
27	0	14	0	13	1
1	1	14	181	194	5
2	1	14	68	68	9
3	1	14	45	43	14
4	1	14	82	87	7
5	1	14	135	150	4
6	1	14	63	60	9
7	1	14	38	47	16
8	1	14	0	17	1
9	1	14	0	21	1
10	1	14	64	64	8
11	1	14	12	4	11
12	1	14	0	3	1
13	1	14	16	23	15
14	1	14	0	2	1
15	1	14	12	3	11
16	1	14	59	63	8
17	1	14	16	12	15
18	1	14	6	16	6
19	1	14	43	48	13
20	1	14	25	21	25
21	1	14	22	3	21
22	1	14	30	29	30
23	1	14	0	8	1
24	1	14	98	98	9
25	1	14	93	80	24
26	1	14	75	63	24
27	1	14	53	24	53
0	2	14	6	26	6
1	2	14	35	31	23
2	2	14	28	32	28
3	2	14	0	1	1
4	2	14	36	44	17
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8	2	14	36	50	25
9	2	14	19	7	18
10	2	14	0	16	1
11	2	14	8	11	8
12	2	14	40	51	21
13	2	14	28	32	28
14	2	14	0	16	1
15	2	14	15	24	14
16	2	14	0	19	1
17	2	14	12	31	11
18	2	14	0	6	1
19	2	14	16	6	15
20	2	14	0	4	1
21	2	14	0	3	1
22	2	14	21	0	20
23	2	14	16	2	15
24	2	14	35	15	34
25	2	14	16	12	15
1	3	14	25	35	25
2	3	14	49	59	13
3	3	14	30	14	29
4	3	14	26	20	26
5	3	14	71	56	10
6	3	14	81	76	9
7	3	14	159	172	5
8	3	14	89	89	8
9	3	14	155	153	5
10	3	14	155	148	5
11	3	14	175	174	5
12	3	14	165	167	5
13	3	14	22	38	21
14	3	14	123	111	8
15	3	14	89	68	10
16	3	14	143	148	6
17	3	14	99	100	8
18	3	14	28	33	28
19	3	14	92	86	8
20	3	14	16	29	15
21	3	14	0	28	1
22	3	14	36	26	35
23	3	14	43	29	24
24	3	14	25	36	24
25	3	14	53	19	53
0	4	14	108	131	8
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2	4	14	25	18	25
3	4	14	0	7	1
4	4	14	36	44	36
5	4	14	57	51	18
6	4	14	150	153	7
7	4	14	40	24	22
8	4	14	36	48	30
9	4	14	30	26	30
10	4	14	67	64	11
11	4	14	47	42	17
12	4	14	96	107	8
13	4	14	0	10	1
14	4	14	100	103	9
15	4	14	28	4	28
16	4	14	43	49	42
17	4	14	37	33	36
18	4	14	57	48	28
19	4	14	38	10	38
20	4	14	122	111	8
21	4	14	65	48	16
22	4	14	67	56	18
1	5	14	16	33	15
2	5	14	0	36	1
3	5	14	68	67	13
4	5	14	0	1	1
5	5	14	0	2	1
6	5	14	0	0	1
7	5	14	23	32	23
8	5	14	0	1	1
9	5	14	26	14	26
10	5	14	77	93	9
11	5	14	18	18	17
12	5	14	24	41	23
13	5	14	0	29	1
14	5	14	54	46	44
15	5	14	109	101	14
16	5	14	20	5	19
17	5	14	83	61	38
18	5	14	79	61	34
19	5	14	71	54	52
20	5	14	72	33	51
0	6	14	25	43	24
1	6	14	0	27	1
2	6	14	13	33	13
3	6	14	0	1	1
4	6	14	0	9	1
5	6	14	22	19	22
6	6	14	54	55	21
7	6	14	0	11	1
8	6	14	0	26	1
9	6	14	0	6	1
10	6	14	0	1	1
11	6	14	0	12	1
12	6	14	40	48	40
15	6	14	35	8	35
16	6	14	61	23	60
1	7	14	0	4	1
2	7	14	0	20	1
3	7	14	70	89	10
4	7	14	24	37	23
5	7	14	0	5	1
1	1	15	71	75	9
2	1	15	0	8	1
3	1	15	149	164	5
4	1	15	74	74	9
5	1	15	12	18	11
6	1	15	87	89	7
7	1	15	25	26	25
8	1	15	79	86	7
9	1	15	8	19	8
10	1	15	30	40	30
11	1	15	45	61	12
12	1	15	0	5	1
13	1	15	35	40	19
14	1	15	0	14	1
15	1	15	0	10	1
16	1	15	30	29	20
17	1	15	52	55	11
18	1	15	19	17	18
19	1	15	17	28	16
20	1	15	16	14	15
21	1	15	55	58	17
22	1	15	35	41	34
0	2	15	15	4	14
1	2	15	74	78	8
2	2	15	34	27	33
3	2	15	35	34	23
4	2	15	75	82	8
5	2	15	31	30	31
6	2	15	0	9	1
7	2	15	0	10	1
8	2	15	27	36	27
9	2	15	0	12	1
10	2	15	0	3	1
11	2	15	91	96	7
12	2	15	39	38	22
13	2	15	82	90	9
14	2	15	55	56	13
15	2	15	30	38	30
16	2	15	34	32	33
17	2	15	0	3	1
18	2	15	13	7	13
19	2	15	27	37	27
20	2	15	0	6	1
21	2	15	0	24	1
22	2	15	71	70	14
23	2	15	20	25	19
1	3	15	0	21	1
2	3	15	44	40	20
3	3	15	154	159	6
4	3	15	21	6	20
5	3	15	39	46	20
6	3	15	27	28	27
7	3	15	10	21	10
8	3	15	88	83	8
9	3	15	180	185	5
10	3	15	65	52	11
11	3	15	103	96	7
12	3	15	0	2	1
13	3	15	0	25	1
14	3	15	0	23	1
15	3	15	107	108	9
16	3	15	38	4	37
17	3	15	146	125	10
18	3	15	85	82	18
19	3	15	58	42	30
20	3	15	0	19	1
21	3	15	30	17	30
0	4	15	97	90	17
1	4	15	135	140	8
2	4	15	56	53	23
3	4	15	55	58	18
4	4	15	20	21	19
5	4	15	48	51	14
6	4	15	39	34	24
7	4	15	92	92	8
8	4	15	36	40	35
9	4	15	49	50	14
10	4	15	74	78	10
11	4	15	8	10	8
12	4	15	26	40	26
13	4	15	0	9	1
14	4	15	0	37	1
15	4	15	29	4	29
16	4	15	65	78	49
1	5	15	57	58	17
2	5	15	12	19	11
3	5	15	74	68	13
4	5	15	40	42	39
5	5	15	70	70	15
6	5	15	0	13	1
7	5	15	15	18	14
8	5	15	18	18	17
9	5	15	30	38	29
10	5	15	0	15	1
11	5	15	27	2	26
12	5	15	78	76	34
13	5	15	0	42	1
0	6	15	0	25	1
1	6	15	0	5	1
2	6	15	0	11	1
3	6	15	0	11	1
4	6	15	0	20	1
5	6	15	0	6	1
6	6	15	41	35	40
7	6	15	0	23	1
0	0	16	82	93	17
1	0	16	114	118	8
2	0	16	69	79	15
3	0	16	135	158	7
4	0	16	108	121	8
5	0	16	119	127	8
6	0	16	68	60	18
7	0	16	32	35	31
8	0	16	29	35	28
9	0	16	0	4	1
10	0	16	0	9	1
11	0	16	38	36	37
12	0	16	20	29	19
13	0	16	0	13	1
14	0	16	22	43	21
15	0	16	22	13	21
16	0	16	0	17	1
17	0	16	0	0	1
18	0	16	0	11	1
19	0	16	16	44	15
20	0	16	22	24	22
1	1	16	24	27	23
2	1	16	0	14	1
3	1	16	0	6	1
4	1	16	10	9	10
5	1	16	0	4	1
6	1	16	59	67	11
7	1	16	20	22	19
8	1	16	25	38	25
9	1	16	6	26	6
10	1	16	0	16	1
11	1	16	20	45	19
12	1	16	0	26	1
13	1	16	17	3	16
14	1	16	0	7	1
15	1	16	0	21	1
16	1	16	0	16	1
17	1	16	0	36	1
18	1	16	0	19	1
19	1	16	0	1	1
20	1	16	0	5	1
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1	2	16	10	22	10
2	2	16	22	36	21
3	2	16	36	47	21
4	2	16	0	14	1
5	2	16	22	44	22
6	2	16	23	16	23
7	2	16	34	41	28
8	2	16	55	53	11
9	2	16	84	97	7
10	2	16	16	20	15
11	2	16	0	26	1
12	2	16	0	8	1
13	2	16	0	14	1
14	2	16	48	63	12
15	2	16	6	19	6
16	2	16	0	8	1

Table 6. . Observed and calculated structure factors for af005a

h	k	l	10Fo	10Fc	10s
17	2	16	22	27	22
18	2	16	0	8	1
19	2	16	48	42	22
1	3	16	30	22	29
2	3	16	67	77	8
3	3	16	62	74	9
4	3	16	30	20	30
5	3	16	49	51	12
6	3	16	94	93	9
7	3	16	32	13	32
8	3	16	47	53	16
9	3	16	16	21	15
10	3	16	0	19	1
11	3	16	40	37	39
12	3	16	15	11	14
13	3	16	0	4	1
14	3	16	0	17	1
15	3	16	0	9	1
16	3	16	0	12	1
0	4	16	35	23	35
1	4	16	32	49	32
2	4	16	33	30	33
3	4	16	37	52	28
4	4	16	38	49	32
5	4	16	0	11	1
6	4	16	45	55	17
7	4	16	22	45	21
8	4	16	0	29	1
9	4	16	0	4	1
10	4	16	0	18	1
11	4	16	59	77	15
12	4	16	0	42	1
13	4	16	43	42	42
1	5	16	33	11	33
2	5	16	35	46	34
3	5	16	0	0	1
4	5	16	30	26	29
5	5	16	0	4	1
6	5	16	16	18	15
7	5	16	0	28	1
8	5	16	36	33	36
9	5	16	54	56	14
10	5	16	0	0	1
11	5	16	25	25	25
1	1	17	41	49	20
2	1	17	0	2	1
3	1	17	23	42	23
4	1	17	0	1	1
5	1	17	0	10	1
6	1	17	23	42	23
7	1	17	15	29	14
8	1	17	28	35	28
9	1	17	0	4	1
10	1	17	0	12	1
11	1	17	0	20	1
12	1	17	0	4	1
13	1	17	21	3	20
14	1	17	0	10	1
15	1	17	13	2	13
0	2	17	26	37	26
1	2	17	0	5	1
2	2	17	10	26	10
3	2	17	77	90	8
4	2	17	44	52	16
5	2	17	0	4	1
6	2	17	24	1	23
7	2	17	92	93	7
8	2	17	46	36	16
9	2	17	100	116	9
10	2	17	28	22	28
11	2	17	35	45	35
12	2	17	16	12	15
13	2	17	98	109	14
14	2	17	8	20	8
15	2	17	39	47	39
1	3	17	0	9	1
2	3	17	0	4	1
3	3	17	0	26	1
4	3	17	29	6	28
5	3	17	0	24	1
6	3	17	0	28	1
7	3	17	0	22	1
8	3	17	54	53	39
9	3	17	10	21	10
10	3	17	0	15	1
11	3	17	0	10	1
12	3	17	29	46	29

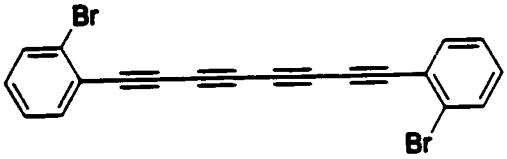

Br
Br

Table 1. Crystal data and structure refinement for af020.

Identification code	af020
Empirical formula	C20 H8 Br2
Formula weight	408.08
Temperature	203(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 3.9243(6) A alpha = 103.008(3) de b = 7.1868(12) A beta = 92.449(3) de c = 14.354(2) A gamma = 95.154(3) deg
Volume	392.01(11) A^3
Z, Calculated density	1, 1.729 Mg/m^3
Absorption coefficient	5.162 mm^-1
F(000)	198
Crystal size	0.22 x 0.18 x 0.07 mm
Theta range for data collection	2.92 to 28.69 deg.
Limiting indices	-5<=h<=5, -9<=k<=9, 0<=l<=19
Reflections collected / unique	3059 / 1771 [R(int) = 0.0406]
Completeness to theta = 28.69	86.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.694436 and 0.241028
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1771 / 0 / 100
Goodness-of-fit on F^2	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0530, wR2 = 0.1137
R indices (all data)	R1 = 0.0637, wR2 = 0.1160
Largest diff. peak and hole	0.755 and -1.090 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af020.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Br	5295(1)	6320(1)	8859(1)	40(1)
C(1)	7364(10)	8262(6)	8314(3)	32(1)
C(2)	8065(11)	10109(7)	8879(3)	37(1)
C(3)	9636(12)	11548(6)	8498(3)	38(1)
C(4)	10567(11)	11136(7)	7560(3)	40(1)
C(5)	9871(12)	9307(7)	6999(3)	40(1)
C(6)	8247(10)	7833(6)	7360(3)	32(1)
C(7)	7491(12)	5948(7)	6763(3)	39(1)
C(8)	6810(13)	4379(7)	6245(3)	41(1)
C(9)	6043(12)	2564(7)	5698(3)	41(1)
C(10)	5375(12)	939(7)	5251(3)	40(1)

Table 3. Bond lengths [A] and angles [deg] for af020.

Br-C(1)	1.887(4)
C(1)-C(2)	1.388(6)
C(1)-C(6)	1.400(6)
C(2)-C(3)	1.385(6)
C(3)-C(4)	1.383(7)
C(4)-C(5)	1.375(7)
C(5)-C(6)	1.397(6)
C(6)-C(7)	1.429(6)
C(7)-C(8)	1.203(7)
C(8)-C(9)	1.363(7)
C(9)-C(10)	1.199(6)
C(10)-C(10)#1	1.378(10)
C(2)-C(1)-C(6)	120.6(4)
C(2)-C(1)-Br	119.0(3)
C(6)-C(1)-Br	120.4(3)
C(3)-C(2)-C(1)	119.9(4)
C(4)-C(3)-C(2)	120.1(4)
C(5)-C(4)-C(3)	120.1(4)
C(4)-C(5)-C(6)	121.1(4)
C(5)-C(6)-C(1)	118.2(4)
C(5)-C(6)-C(7)	120.4(4)
C(1)-C(6)-C(7)	121.4(4)
C(8)-C(7)-C(6)	178.5(5)
C(7)-C(8)-C(9)	177.2(5)
C(10)-C(9)-C(8)	177.3(5)
C(9)-C(10)-C(10)#1	179.2(6)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z+1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af020. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Br	39(1)	37(1)	48(1)	16(1)	10(1)	1(1)
C(1)	29(2)	34(2)	35(2)	11(2)	6(2)	7(2)
C(2)	39(2)	37(2)	35(2)	8(2)	8(2)	7(2)
C(3)	41(2)	28(2)	44(3)	6(2)	-3(2)	0(2)
C(4)	35(2)	41(3)	48(3)	20(2)	1(2)	-4(2)
C(5)	41(2)	49(3)	34(2)	17(2)	4(2)	3(2)
C(6)	30(2)	32(2)	34(2)	8(2)	-1(2)	4(2)
C(7)	40(2)	43(3)	34(2)	11(2)	3(2)	2(2)
C(8)	47(3)	44(3)	31(2)	6(2)	3(2)	3(2)
C(9)	45(3)	46(3)	31(2)	9(2)	3(2)	0(2)
C(10)	47(3)	44(3)	29(2)	7(2)	6(2)	2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af020.

	x	y	z	U(eq)
H(2A)	7474	10383	9520	44
H(3A)	10071	12804	8876	46
H(4A)	11676	12108	7307	48
H(5A)	10499	9045	6361	48

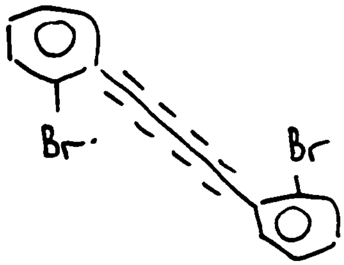
Table 6. Torsion angles [deg] for af020.

C(6)-C(1)-C(2)-C(3)	0.3(6)
Br-C(1)-C(2)-C(3)	178.9(3)
C(1)-C(2)-C(3)-C(4)	-1.3(6)
C(2)-C(3)-C(4)-C(5)	1.3(7)
C(3)-C(4)-C(5)-C(6)	-0.5(6)
C(4)-C(5)-C(6)-C(1)	-0.4(6)
C(4)-C(5)-C(6)-C(7)	178.9(4)
C(2)-C(1)-C(6)-C(5)	0.5(6)
Br-C(1)-C(6)-C(5)	-178.0(3)
C(2)-C(1)-C(6)-C(7)	-178.9(4)
Br-C(1)-C(6)-C(7)	2.6(5)
C(5)-C(6)-C(7)-C(8)	-59(20)
C(1)-C(6)-C(7)-C(8)	121(20)
C(6)-C(7)-C(8)-C(9)	-147(16)
C(7)-C(8)-C(9)-C(10)	-1(19)
C(8)-C(9)-C(10)-C(10)#1	-17(54)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z+1

Shawn.

Dr. Fallis



$C_{20}H_8Br_2$.

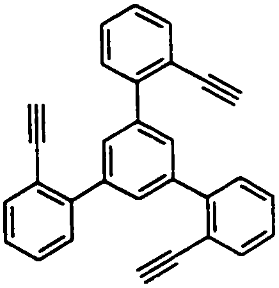


Table 1. Crystal data and structure refinement for af011.

Identification code	af011	
Empirical formula	C30 H18	
Formula weight	378.44	
Temperature	203(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 11.650(2) A	alpha = 71.585(4) deg
	b = 14.144(3) A	beta = 68.153(4) deg
	c = 15.046(3) A	gamma = 71.384(4) deg
Volume	2125.3(7) A^3	
Z, Calculated density	4, 1.183 Mg/m^3	
Absorption coefficient	0.067 mm^-1	
F(000)	792	
Crystal size	0.15 x 0.15 x 0.04 mm	
Theta range for data collection	1.56 to 20.81 deg.	
Limiting indices	-10<=h<=11, -13<=k<=14, 0<=l<=15	
Reflections collected / unique	16532 / 4427 [R(int) = 0.1266]	
Completeness to theta = 20.81	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.928079 and 0.513091	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4427 / 0 / 542	
Goodness-of-fit on F^2	1.060	
Final R indices [I>2sigma(I)]	R1 = 0.0599, wR2 = 0.1069	
R indices (all data)	R1 = 0.1166, wR2 = 0.1163	
Extinction coefficient	0.0095(9)	
Largest diff. peak and hole	0.228 and -0.223 e.A^-3	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af011.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2478(4)	992(4)	2099(4)	34(1)
C(2)	2162(5)	1699(4)	1298(4)	37(1)
C(3)	1031(5)	2430(4)	1388(4)	37(1)
C(4)	187(5)	2440(4)	2316(4)	37(1)
C(5)	446(4)	1743(4)	3147(4)	33(1)
C(6)	1592(5)	1030(4)	3014(4)	37(1)
C(7)	3621(5)	133(4)	1974(4)	34(1)
C(8)	3897(5)	-412(4)	1265(4)	41(1)
C(9)	4912(5)	-1256(4)	1156(4)	48(2)
C(10)	5660(5)	-1580(4)	1755(4)	49(2)
C(11)	5461(5)	-1038(4)	2449(4)	50(2)
C(12)	4446(5)	-180(4)	2567(4)	41(2)
C(13)	4301(5)	368(5)	3251(5)	55(2)
C(14)	4231(6)	789(5)	3820(5)	77(2)
C(15)	653(5)	3129(4)	509(4)	35(1)
C(16)	-522(5)	3223(4)	458(4)	42(2)
C(17)	-942(5)	3877(4)	-333(5)	57(2)
C(18)	-124(6)	4444(4)	-1084(4)	59(2)
C(19)	1037(6)	4360(4)	-1055(4)	48(2)
C(20)	1457(5)	3712(4)	-260(4)	45(2)
C(21)	2649(7)	3714(5)	-218(4)	60(2)
C(22)	3643(7)	3774(5)	-244(5)	79(2)
C(23)	-497(5)	1755(4)	4123(4)	38(1)
C(24)	-147(5)	1823(4)	4879(4)	49(2)
C(25)	-1029(6)	1856(4)	5813(4)	57(2)
C(26)	-2244(6)	1801(4)	5990(4)	54(2)
C(27)	-2609(5)	1710(4)	5251(4)	49(2)
C(28)	-1723(5)	1657(3)	4319(4)	37(1)
C(29)	-2140(5)	1522(4)	3581(4)	47(2)
C(30)	-2482(5)	1406(5)	2985(5)	68(2)
C(31)	8537(5)	2384(4)	8282(3)	37(1)
C(32)	7403(5)	2999(4)	8150(3)	40(2)
C(33)	7370(5)	3914(4)	7448(4)	36(1)
C(34)	8502(5)	4199(4)	6869(3)	39(1)
C(35)	9652(5)	3598(4)	6993(4)	35(1)
C(36)	9669(5)	2694(4)	7712(4)	38(1)
C(37)	8587(5)	1351(4)	8978(4)	38(1)
C(38)	9440(5)	514(4)	8608(4)	48(2)
C(39)	9477(6)	-465(5)	9201(5)	65(2)
C(40)	8692(7)	-612(5)	10128(5)	63(2)
C(41)	7850(6)	190(5)	10520(4)	61(2)
C(42)	7800(5)	1210(5)	9918(4)	48(2)
C(43)	6920(6)	2011(5)	10370(4)	62(2)
C(44)	6166(7)	2605(6)	10821(4)	98(3)
C(45)	6151(5)	4567(4)	7282(3)	37(1)
C(46)	5295(5)	4116(4)	7217(3)	43(2)
C(47)	4192(5)	4674(5)	7043(4)	54(2)
C(48)	3864(5)	5718(5)	6952(4)	58(2)
C(49)	4664(6)	6187(4)	7031(4)	55(2)

C(50)	5790(5)	5618(5)	7225(4)	42(2)
C(51)	6569(6)	6151(5)	7337(4)	53(2)
C(52)	7135(6)	6614(5)	7459(5)	76(2)
C(53)	10860(5)	3939(4)	6375(3)	39(1)
C(54)	10865(5)	4966(5)	6161(4)	50(2)
C(55)	11944(6)	5317(4)	5589(4)	54(2)
C(56)	13067(6)	4656(5)	5218(4)	61(2)
C(57)	13075(5)	3636(5)	5420(4)	54(2)
C(58)	11974(5)	3277(5)	5977(4)	42(2)
C(59)	12074(5)	2214(6)	6097(4)	54(2)
C(60)	12200(5)	1330(5)	6169(4)	71(2)

Table 3. Bond lengths [A] and angles [deg] for af011.

C(1)-C(6)	1.384(6)
C(1)-C(2)	1.387(6)
C(1)-C(7)	1.487(6)
C(2)-C(3)	1.382(6)
C(3)-C(4)	1.379(6)
C(3)-C(15)	1.500(6)
C(4)-C(5)	1.392(6)
C(5)-C(6)	1.381(6)
C(5)-C(23)	1.472(6)
C(7)-C(8)	1.394(6)
C(7)-C(12)	1.426(6)
C(8)-C(9)	1.388(6)
C(9)-C(10)	1.359(6)
C(10)-C(11)	1.394(6)
C(11)-C(12)	1.403(6)
C(12)-C(13)	1.406(7)
C(13)-C(14)	1.155(7)
C(15)-C(16)	1.361(6)
C(15)-C(20)	1.406(6)
C(16)-C(17)	1.400(6)
C(17)-C(18)	1.394(7)
C(18)-C(19)	1.336(7)
C(19)-C(20)	1.403(6)
C(20)-C(21)	1.414(8)
C(21)-C(22)	1.173(7)
C(23)-C(24)	1.382(6)
C(23)-C(28)	1.389(6)
C(24)-C(25)	1.403(6)
C(25)-C(26)	1.362(6)
C(26)-C(27)	1.384(6)
C(27)-C(28)	1.405(6)
C(28)-C(29)	1.450(7)
C(29)-C(30)	1.179(7)
C(31)-C(32)	1.377(6)
C(31)-C(36)	1.398(6)
C(31)-C(37)	1.503(6)
C(32)-C(33)	1.388(6)
C(33)-C(34)	1.387(6)
C(33)-C(45)	1.488(6)
C(34)-C(35)	1.382(6)
C(35)-C(36)	1.390(6)
C(35)-C(53)	1.502(6)
C(37)-C(42)	1.361(6)
C(37)-C(38)	1.396(6)
C(38)-C(39)	1.388(7)
C(39)-C(40)	1.348(7)
C(40)-C(41)	1.370(7)
C(41)-C(42)	1.437(7)
C(42)-C(43)	1.423(8)
C(43)-C(44)	1.175(7)
C(45)-C(46)	1.387(6)
C(45)-C(50)	1.393(6)
C(46)-C(47)	1.348(6)
C(47)-C(48)	1.377(7)
C(48)-C(49)	1.360(7)
C(49)-C(50)	1.384(6)

C(50)-C(51)	1.432(8)
C(51)-C(52)	1.157(7)
C(53)-C(58)	1.386(6)
C(53)-C(54)	1.387(6)
C(54)-C(55)	1.375(6)
C(55)-C(56)	1.379(7)
C(56)-C(57)	1.376(7)
C(57)-C(58)	1.397(7)
C(58)-C(59)	1.428(8)
C(59)-C(60)	1.185(7)

C(6)-C(1)-C(2)	117.1(5)
C(6)-C(1)-C(7)	120.7(5)
C(2)-C(1)-C(7)	121.5(5)
C(1)-C(2)-C(3)	122.7(5)
C(4)-C(3)-C(2)	117.9(5)
C(4)-C(3)-C(15)	119.8(5)
C(2)-C(3)-C(15)	122.0(5)
C(3)-C(4)-C(5)	122.0(5)
C(6)-C(5)-C(4)	117.7(5)
C(6)-C(5)-C(23)	121.9(5)
C(4)-C(5)-C(23)	120.4(5)
C(5)-C(6)-C(1)	122.7(5)
C(8)-C(7)-C(12)	117.8(5)
C(8)-C(7)-C(1)	119.4(5)
C(12)-C(7)-C(1)	122.8(5)
C(7)-C(8)-C(9)	121.6(5)
C(10)-C(9)-C(8)	120.3(5)
C(9)-C(10)-C(11)	120.5(5)
C(10)-C(11)-C(12)	120.1(5)
C(11)-C(12)-C(13)	118.4(5)
C(11)-C(12)-C(7)	119.5(5)
C(13)-C(12)-C(7)	122.0(5)
C(14)-C(13)-C(12)	177.0(7)
C(16)-C(15)-C(20)	118.5(5)
C(16)-C(15)-C(3)	119.0(5)
C(20)-C(15)-C(3)	122.5(5)
C(15)-C(16)-C(17)	121.8(5)
C(18)-C(17)-C(16)	118.5(6)
C(19)-C(18)-C(17)	120.8(6)
C(18)-C(19)-C(20)	120.9(6)
C(19)-C(20)-C(15)	119.5(5)
C(19)-C(20)-C(21)	118.9(6)
C(15)-C(20)-C(21)	121.5(5)
C(22)-C(21)-C(20)	175.1(7)
C(24)-C(23)-C(28)	118.7(5)
C(24)-C(23)-C(5)	119.5(5)
C(28)-C(23)-C(5)	121.8(5)
C(23)-C(24)-C(25)	120.9(5)
C(26)-C(25)-C(24)	120.1(6)
C(25)-C(26)-C(27)	120.0(5)
C(26)-C(27)-C(28)	120.1(5)
C(27)-C(28)-C(23)	120.1(5)
C(27)-C(28)-C(29)	118.0(5)
C(23)-C(28)-C(29)	122.0(5)
C(30)-C(29)-C(28)	179.5(7)
C(32)-C(31)-C(36)	119.5(5)
C(32)-C(31)-C(37)	121.0(5)
C(36)-C(31)-C(37)	119.4(5)
C(31)-C(32)-C(33)	120.9(5)

C(34)-C(33)-C(32)	119.1(5)
C(34)-C(33)-C(45)	119.8(5)
C(32)-C(33)-C(45)	121.1(5)
C(33)-C(34)-C(35)	121.1(5)
C(34)-C(35)-C(36)	119.2(5)
C(34)-C(35)-C(53)	120.0(5)
C(36)-C(35)-C(53)	120.7(5)
C(35)-C(36)-C(31)	120.2(5)
C(42)-C(37)-C(38)	120.3(5)
C(42)-C(37)-C(31)	122.4(5)
C(38)-C(37)-C(31)	117.2(5)
C(39)-C(38)-C(37)	119.7(6)
C(40)-C(39)-C(38)	120.3(6)
C(39)-C(40)-C(41)	121.6(6)
C(40)-C(41)-C(42)	118.8(6)
C(37)-C(42)-C(43)	124.6(6)
C(37)-C(42)-C(41)	119.2(6)
C(43)-C(42)-C(41)	116.2(6)
C(44)-C(43)-C(42)	173.8(7)
C(46)-C(45)-C(50)	117.4(5)
C(46)-C(45)-C(33)	119.4(5)
C(50)-C(45)-C(33)	123.2(5)
C(47)-C(46)-C(45)	121.9(5)
C(46)-C(47)-C(48)	120.2(6)
C(49)-C(48)-C(47)	119.7(6)
C(48)-C(49)-C(50)	120.4(6)
C(49)-C(50)-C(45)	120.2(6)
C(49)-C(50)-C(51)	117.8(6)
C(45)-C(50)-C(51)	122.0(5)
C(52)-C(51)-C(50)	176.0(7)
C(58)-C(53)-C(54)	117.8(5)
C(58)-C(53)-C(35)	123.0(5)
C(54)-C(53)-C(35)	119.2(5)
C(55)-C(54)-C(53)	121.3(5)
C(56)-C(55)-C(54)	121.1(6)
C(55)-C(56)-C(57)	118.2(6)
C(56)-C(57)-C(58)	121.1(6)
C(53)-C(58)-C(57)	120.4(6)
C(53)-C(58)-C(59)	123.3(5)
C(57)-C(58)-C(59)	116.3(5)
C(60)-C(59)-C(58)	177.5(7)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af011. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	31(3)	27(4)	43(4)	-13(3)	-5(3)	-7(3)
C(2)	44(4)	30(4)	37(4)	-10(3)	-4(3)	-18(3)
C(3)	44(4)	20(3)	44(4)	-9(3)	-12(3)	-2(3)
C(4)	41(3)	26(3)	42(4)	-12(3)	-8(3)	-6(3)
C(5)	36(3)	26(3)	41(4)	-13(3)	-15(3)	-3(3)
C(6)	42(3)	28(3)	43(4)	-7(3)	-11(3)	-13(3)
C(7)	40(3)	21(3)	42(4)	-4(3)	-7(3)	-16(3)
C(8)	43(3)	34(4)	44(4)	-9(3)	-7(3)	-13(3)
C(9)	50(4)	39(4)	47(4)	-16(3)	3(3)	-16(3)
C(10)	40(4)	23(4)	72(4)	-12(4)	-7(3)	-2(3)
C(11)	47(4)	34(4)	60(4)	5(3)	-19(3)	-11(3)
C(12)	41(4)	39(4)	43(4)	-4(3)	-12(3)	-16(3)
C(13)	61(4)	54(5)	51(4)	-17(4)	-17(4)	-9(4)
C(14)	72(5)	79(6)	95(6)	-36(5)	-35(5)	-8(4)
C(15)	37(4)	24(3)	42(4)	-15(3)	-4(3)	-7(3)
C(16)	52(4)	25(4)	52(4)	-12(3)	-19(3)	-5(3)
C(17)	64(4)	43(4)	72(5)	-19(4)	-25(4)	-11(4)
C(18)	88(5)	38(4)	53(4)	-16(4)	-34(4)	2(4)
C(19)	67(4)	30(4)	44(4)	-2(3)	-23(3)	-5(3)
C(20)	47(4)	32(4)	49(4)	-24(3)	-7(4)	3(3)
C(21)	59(5)	50(4)	53(4)	-5(3)	0(4)	-15(5)
C(22)	67(5)	75(5)	83(5)	-18(4)	-5(4)	-20(5)
C(23)	45(4)	32(4)	37(4)	-5(3)	-14(3)	-11(3)
C(24)	41(3)	47(4)	53(4)	-8(3)	-7(4)	-11(3)
C(25)	72(5)	44(4)	49(4)	-2(3)	-14(4)	-18(4)
C(26)	68(5)	33(4)	45(4)	-5(3)	-1(4)	-11(4)
C(27)	37(3)	32(4)	65(4)	-8(3)	-3(4)	-11(3)
C(28)	41(4)	18(3)	40(4)	-3(3)	-7(3)	-3(3)
C(29)	45(4)	40(4)	54(4)	-10(4)	-20(3)	-3(3)
C(30)	73(5)	55(5)	83(5)	-20(4)	-36(4)	-4(4)
C(31)	41(3)	31(4)	36(3)	-1(3)	-15(3)	-6(3)
C(32)	47(4)	31(4)	42(4)	0(3)	-17(3)	-12(3)
C(33)	36(3)	36(4)	36(3)	-7(3)	-11(3)	-10(3)
C(34)	51(4)	30(3)	38(3)	-6(3)	-20(3)	-4(3)
C(35)	41(4)	31(4)	39(4)	-10(3)	-18(3)	-8(3)
C(36)	41(4)	29(4)	42(3)	-8(3)	-17(3)	1(3)
C(37)	39(3)	39(4)	41(4)	-7(3)	-15(3)	-14(3)
C(38)	57(4)	30(4)	69(4)	-6(4)	-37(3)	-10(3)
C(39)	68(5)	46(5)	84(5)	-10(4)	-34(4)	-10(4)
C(40)	91(5)	33(4)	80(6)	2(4)	-48(5)	-20(4)
C(41)	71(5)	69(5)	52(4)	11(4)	-26(4)	-44(4)
C(42)	54(4)	47(4)	47(4)	-14(4)	-17(3)	-11(4)
C(43)	67(5)	80(6)	37(4)	1(4)	-13(4)	-31(4)
C(44)	98(6)	118(7)	55(5)	-39(5)	-5(4)	2(5)
C(45)	37(3)	39(4)	38(3)	-5(3)	-14(3)	-16(3)
C(46)	40(3)	36(4)	48(4)	-4(3)	-18(3)	-4(3)
C(47)	47(4)	60(5)	59(4)	-8(4)	-20(3)	-19(4)
C(48)	45(4)	53(5)	68(4)	-5(4)	-23(3)	-2(4)
C(49)	52(4)	45(4)	66(4)	-2(3)	-21(3)	-16(4)
C(50)	45(4)	34(4)	43(4)	-7(3)	-11(3)	-7(3)

C(51)	62(5)	28(4)	66(4)	-10(4)	-18(4)	-8(3)
C(52)	83(5)	50(5)	111(6)	-31(5)	-38(4)	-13(4)
C(53)	38(4)	39(4)	38(4)	-2(3)	-15(3)	-9(3)
C(54)	47(4)	52(5)	50(4)	-3(3)	-16(3)	-17(3)
C(55)	68(4)	40(4)	56(4)	8(3)	-23(4)	-30(4)
C(56)	60(5)	76(5)	42(4)	-3(4)	-8(3)	-30(4)
C(57)	44(4)	67(5)	47(4)	-15(4)	-11(3)	-11(4)
C(58)	42(4)	50(4)	35(4)	-7(3)	-5(3)	-23(4)
C(59)	56(4)	56(5)	46(4)	-10(4)	-12(3)	-14(4)
C(60)	73(5)	58(5)	80(5)	-23(5)	-22(4)	-6(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af011.

	x	y	z	U(eq)
H(2A)	2741	1680	667	44
H(4A)	-587	2933	2391	44
H(6A)	1778	551	3568	45
H(8A)	3384	-203	850	49
H(9A)	5083	-1605	666	57
H(10A)	6316	-2174	1700	59
H(11A)	6007	-1248	2838	59
H(14)	4174	1132	4282	93
H(16A)	-1067	2837	968	50
H(17A)	-1756	3932	-357	69
H(18A)	-393	4892	-1618	70
H(19A)	1581	4740	-1574	58
H(22)	4439	3822	-265	95
H(24A)	693	1849	4766	59
H(25A)	-781	1915	6317	68
H(26A)	-2835	1825	6616	65
H(27A)	-3451	1683	5371	58
H(30)	-2755	1314	2510	82
H(32A)	6640	2797	8541	48
H(34A)	8488	4811	6383	47
H(36A)	10443	2290	7815	45
H(38A)	9986	614	7960	58
H(39A)	10053	-1028	8954	77
H(40A)	8722	-1281	10517	76
H(41A)	7315	75	11171	73
H(44)	5564	3079	11182	118
H(46A)	5491	3402	7298	51
H(47A)	3644	4349	6982	65
H(48A)	3090	6104	6836	69
H(49A)	4451	6901	6953	66
H(52)	7594	6990	7558	91
H(54A)	10116	5432	6413	60
H(55A)	11916	6019	5448	64
H(56A)	13807	4897	4838	73
H(57A)	13833	3173	5178	64
H(60)	12300	629	6226	85

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
1	0	0	694	687	10
2	0	0	29	0	29
3	0	0	87	78	15
4	0	0	138	159	7
5	0	0	286	290	6
6	0	0	296	299	7
7	0	0	0	21	1
8	0	0	0	22	1
9	0	0	26	28	25
10	0	0	63	83	28
-10	1	0	59	65	29
-9	1	0	101	92	17
-8	1	0	106	101	16
-7	1	0	87	59	21
-6	1	0	76	87	18
-5	1	0	172	185	8
-4	1	0	240	233	6
-3	1	0	281	305	6
-2	1	0	322	337	5
-1	1	0	170	166	5
0	1	0	10	5	10
2	1	0	67	54	6
3	1	0	444	484	7
4	1	0	415	419	7
5	1	0	363	376	7
6	1	0	0	27	1
7	1	0	45	41	45
8	1	0	66	101	26
9	1	0	240	245	8
10	1	0	181	154	8
-10	2	0	0	9	1
-9	2	0	104	107	14
-8	2	0	45	18	45
-7	2	0	111	105	15
-6	2	0	88	80	12
-5	2	0	323	330	6
-4	2	0	685	698	10
-3	2	0	260	253	6
-2	2	0	197	201	6
-1	2	0	1228	1220	16
0	2	0	141	149	6
1	2	0	374	348	6
2	2	0	370	348	7
3	2	0	513	532	9
4	2	0	45	54	21
5	2	0	163	160	5
6	2	0	121	115	7
7	2	0	20	40	19
8	2	0	83	64	14
9	2	0	136	119	10
10	2	0	0	28	1
-9	3	0	84	104	19
-8	3	0	24	28	24
-7	3	0	10	31	10
-6	3	0	121	146	11
-5	3	0	241	248	7
-4	3	0	106	83	11
-3	3	0	498	501	8
-2	3	0	27	12	26
-1	3	0	46	63	32
0	3	0	425	450	22
1	3	0	782	684	10
2	3	0	507	514	9
3	3	0	194	205	8
4	3	0	267	278	6
5	3	0	126	122	9
6	3	0	205	210	5
7	3	0	0	4	1
8	3	0	140	133	9
9	3	0	31	41	31
10	3	0	177	169	10
-9	4	0	56	82	27
-8	4	0	35	2	35
-7	4	0	0	27	1
-6	4	0	44	54	44
-5	4	0	0	43	1
-4	4	0	421	394	6
-3	4	0	180	183	6
-2	4	0	559	556	8
-1	4	0	627	638	8
0	4	0	709	753	11
1	4	0	417	419	6
2	4	0	227	152	43
3	4	0	672	566	12
4	4	0	226	213	8
5	4	0	425	356	11
6	4	0	84	71	22
7	4	0	58	28	58
8	4	0	113	109	21
9	4	0	85	51	36
10	4	0	207	204	14
-8	5	0	32	13	31
-7	5	0	31	34	31
-6	5	0	34	19	33
-5	5	0	112	110	15
-4	5	0	51	64	24
-3	5	0	163	153	6
-2	5	0	279	282	6
-1	5	0	26	11	26
0	5	0	523	516	9
1	5	0	0	3	1
2	5	0	385	330	10
3	5	0	72	85	23
4	5	0	193	171	13
5	5	0	288	259	9
6	5	0	182	189	9
7	5	0	0	4	1
8	5	0	57	68	56
9	5	0	81	65	41
10	5	0	86	83	39
-8	6	0	71	116	29
-7	6	0	109	117	22
-6	6	0	50	39	49
-5	6	0	81	75	21
-4	6	0	251	252	6
-3	6	0	3	27	3
-2	6	0	211	216	6
-1	6	0	228	238	6
0	6	0	230	239	6
1	6	0	77	67	16
2	6	0	370	342	6
3	6	0	234	243	7
4	6	0	271	260	7
5	6	0	313	301	9
6	6	0	235	249	9
7	6	0	146	130	16
8	6	0	193	184	16
9	6	0	98	93	44
10	6	0	0	23	1
-7	7	0	9	7	9
-6	7	0	122	113	21
-5	7	0	154	141	12
-4	7	0	21	4	20
-3	7	0	76	71	20
-2	7	0	49	65	37
-1	7	0	333	336	6
0	7	0	171	163	8
1	7	0	44	19	43
2	7	0	223	65	38
3	7	0	208	198	9
4	7	0	140	130	12
5	7	0	192	184	10
6	7	0	74	78	39
7	7	0	113	113	23
8	7	0	87	85	46
9	7	0	84	81	54
10	7	0	96	70	52
-6	8	0	135	160	16
-5	8	0	147	166	18
-4	8	0	93	79	20
-3	8	0	0	5	1
-2	8	0	149	161	9
-1	8	0	477	483	8
0	8	0	13	5	13
1	8	0	230	211	10
2	8	0	199	100	20
3	8	0	296	275	8
4	8	0	135	140	14
5	8	0	21	3	20
6	8	0	48	18	48
7	8	0	0	27	1
8	8	0	170	166	20
9	8	0	160	140	24
-6	9	0	0	17	1
-5	9	0	38	30	37
-4	9	0	50	65	49
-3	9	0	70	58	70
-2	9	0	122	107	14
-1	9	0	108	93	17
0	9	0	170	169	10
1	9	0	199	203	11
2	9	0	72	72	33
3	9	0	50	68	50
4	9	0	221	224	10
5	9	0	130	123	18
6	9	0	238	242	10
7	9	0	70	71	39
8	9	0	41	25	41
9	9	0	71	29	71
-5	10	0	17	45	17
-4	10	0	90	84	30
-3	10	0	52	63	52
-2	10	0	31	14	31
-1	10	0	110	87	17
0	10	0	125	125	15
1	10	0	70	80	25
2	10	0	178	180	11
3	10	0	0	27	1
4	10	0	0	11	1
5	10	0	250	267	10
6	10	0	188	158	14
7	10	0	112	112	20
8	10	0	0	30	1
-3	11	0	110	108	31
-2	11	0	51	28	50
-1	11	0	62	20	61
0	11	0	73	55	35
1	11	0	47	26	46
2	11	0	0	9	1
3	11	0	155	124	17
4	11	0	35	19	35
5	11	0	234	235	10
6	11	0	0	42	1
7	11	0	38	40	38
8	11	0	83	43	31
-2	12	0	0	14	1
-1	12	0	116	136	17
0	12	0	0	23	1
1	12	0	71	73	41
2	12	0	12	44	11
3	12	0	60	42	59
4	12	0	105	95	23
5	12	0	34	75	33
6	12	0	87	91	27
0	13	0	0	68	1
1	13	0	0	9	1
2	13	0	39	50	39
3	13	0	72	44	30
4	13	0	94	82	24
5	13	0	47	8	47
-3	-13	1	84	116	27
-2	-13	1	0	10	1
-1	-13	1	101	137	30
-6	-12	1	86	98	31
-5	-12	1	30	13	30
-4	-12	1	145	137	17
-3	-12	1	111	92	21
-2	-12	1	59	74	58
-1	-12	1	76	107	33
0	-12	1	104	103	23
1	-12	1	79	78	35
2	-12	1	60	75	59
-7	-11	1	95	125	26
-6	-11	1	244	235	11
-5	-11	1	133	97	20
-4	-11	1	45	53	45
-3	-11	1	132	139	19
-2	-11	1	46	35	46
-1	-11	1	98	102	22
0	-11	1	224	231	12
1	-11	1	255	258	10
2	-11	1	161	157	14
3	-11	1	41	6	40
-8	-10	1	63	61	62
-7	-10	1	25	10	24
-6	-10	1	0	28	1
-5	-10	1	86	65	37
-4	-10	1	186	191	14
-3	-10	1	100	110	25
-2	-10	1	61	66	34
-1	-10	1	219	222	11
0	-10	1	104	93	20
1	-10	1	383	374	10
2	-10	1	180	183	13
3	-10	1	396	420	12
4	-10	1	237	249	14
5	-10	1	0	33	1
-8	-9	1	68	65	54
-7	-9	1	135	132	18
-6	-9	1	141	114	16
-5	-9	1	218	209	10
-4	-9	1	122	119	18
-3	-9	1	92	97	22
-2	-9	1	33	28	32
-1	-9	1	177	187	10
0	-9	1	40	81	39
1	-9	1	99	80	22
2	-9	1	41	56	41
3	-9	1	116	85	18
4	-9	1	176	190	15
5	-9	1	24	37	24
6	-9	1	95	87	27
-9	-8	1	112	130	33
-8	-8	1	102	96	37
-7	-8	1	122	116	27
-6	-8	1	92	108	36
-5	-8	1	55	38	54
-4	-8	1	0	12	1
-3	-8	1	80	35	25
-2	-8	1	0	22	1
-1	-8	1	272	274	7
0	-8	1	146	146	11
1	-8	1	83	74	21
2	-8	1	111	117	15
3	-8	1	161	158	10
4	-8	1	152	167	18
6	-8	1	66	77	66
7	-8	1	0	5	1
-9	-7	1	63	45	63
-8	-7	1	37	89	37
-7	-7	1	61	48	60
-6	-7	1	103	108	26
-5	-7	1	168	134	18
-4	-7	1	160	148	12
-3	-7	1	60	65	36
-2	-7	1	227	217	8
-1	-7	1	68	84	21
0	-7	1	72	73	21
1	-7	1	282	273	7
2	-7	1	360	358	7
3	-7	1	94	76	15
4	-7	1	133	128	10
5	-7	1	0	10	1
6	-7	1	90	112	31
7	-7	1	91	112	24
-10	-6	1	68	87	68
-9	-6	1	130	134	27
-8	-6	1	169	166	25
-7	-6	1	0	41	1
-6	-6	1	157	164	15
-5	-6	1	0	20	1
-4	-6	1	62	95	61
-3	-6	1	19	17	19
-2	-6	1	62	41	23
-1	-6	1	719	743	10
0	-6	1	285	281	6
1	-6	1	229	223	6
2	-6	1	379	394	6
3	-6	1	0	34	1
4	-6	1	146	143	8
5	-6	1	37	24	37
6	-6	1	39	27	39
7	-6	1	67	63	39
8	-6	1	74	71	35
-10	-5	1	125	126	26
-9	-5	1	229	137	28
-8	-5	1	0	17	1
-7	-5	1	115	101	18
-6	-5	1	54	46	54
-5	-5	1	216	217	9
-4	-5	1	425	353	12
-3	-5	1	79	58	42
-2	-5	1	506	479	8
-1	-5	1	41	53	30
0	-5	1	282	287	6
1	-5	1	637	637	9
2	-5	1	204	214	6
3	-5	1	147	139	7
4	-5	1	44	48	44
5	-5	1	102	95	15
6	-5	1	51	58	50
7	-5	1	60	28	60
8	-5	1	21	9	21
-10	-4	1	41	0	40
-9	-4	1	49	32	49
-8	-4	1	61	30	60
-7	-4	1	70	57	31
-6	-4	1	171	142	13
-5	-4	1	158	159	10
-4	-4	1	474	471	9
-3	-4	1	458	279	32
-2	-4	1	107	76	16
-1	-4	1	447	485	15
0	-4	1	266	252	6
1	-4	1	454	449	7
2	-4	1	32	6	31
3	-4	1	210	202	5
4	-4	1	221	170	14
5	-4	1	238	227	6
6	-4	1	130	140	11
7	-4	1	72	57	28
8	-4	1	24	37	24
9	-4	1	0	4	1
-10	-3	1	47	77	46
-9	-3	1	202	200	7
-8	-3	1	42	41	41
-7	-3	1	115	109	15
-6	-3	1	128	134	12
-5	-3	1	605	590	8
-4	-3	1	129	107	12
-3	-3	1	416	364	10
-2	-3	1	968	871	15
-1	-3	1	674	658	11
0	-3	1	837	793	11
1	-3	1	105	106	11
2	-3	1	164	165	7
3	-3	1	77	69	12
4	-3	1	93	79	14
5	-3	1	99	85	11
6	-3	1	145	146	8
7	-3	1	124	108	12
8	-3	1	23	12	22
9	-3	1	17	5	16
-10	-2	1	103	101	12
-9	-2	1	36	25	36
-8	-2	1	134	139	9
-7	-2	1	56	45	20
-6	-2	1	18	20	18
-5	-2	1	63	60	13
-4	-2	1	159	163	4
-3	-2	1	242	238	6
-2	-2	1	1063	1027	14
-1	-2	1	1107	1051	17
0	-2	1	468	473	6
1	-2	1	1091	1085	14
2	-2	1	370	385	6
3	-2	1	211	217	6
4	-2	1	893	854	12
5	-2	1	386	365	6
6	-2	1	50	54	23
7	-2	1	78	89	15
8	-2	1	54	86	36
9	-2	1	85	97	18
10	-2	1	31	9	31
-10	-1	1	96	86	14
-9	-1	1	147	135	9
-8	-1	1	121	118	11
-7	-1	1	113	102	10
-6	-1	1	112	104	8
-5	-1	1	125	112	7
-4	-1	1	91	79	8
-3	-1	1	589	639	6
-2	-1	1	392	412	4
-1	-1	1	72	84	8
0	-1	1	206	213	4
1	-1	1	300	324	5
2	-1	1	19	5	18
3	-1	1	225	222	5
4	-1	1	10	27	9
5	-1	1	127	130	7
6	-1	1	0	1	1
7	-1	1	137	147	8
8	-1	1	46	53	45
9	-1	1	122	130	12
10	-1	1	76	70	22
-10	0	1	53	49	31
-9	0	1	161	178	10
-8	0	1	329	352	8
-7	0	1	0	7	1
-6	0	1	28	27	28
-5	0	1	129	130	10
-4	0	1	36	0	36
-3	0	1	316	327	6
-2	0	1	320	350	5
-1	0	1	734	774	7

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
1	0	1	342	369	6
2	0	1	266	286	25
3	0	1	212	210	6
4	0	1	496	518	8
5	0	1	341	323	7
6	0	1	16	42	16
7	0	1	66	74	26
8	0	1	29	56	29
9	0	1	285	277	9
10	0	1	103	112	15
-10	1	1	0	0	1
-9	1	1	0	24	1
-8	1	1	49	37	48
-7	1	1	129	120	15
-6	1	1	82	97	18
-5	1	1	0	29	1
-4	1	1	64	62	21
-3	1	1	158	159	8
-2	1	1	222	215	6
-1	1	1	4	12	4
0	1	1	486	509	7
1	1	1	425	462	11
3	1	1	898	915	13
4	1	1	77	76	14
5	1	1	183	180	6
6	1	1	56	61	33
7	1	1	88	88	18
8	1	1	7	0	6
9	1	1	18	40	18
10	1	1	93	87	15
-9	2	1	28	14	27
-8	2	1	47	24	47
-7	2	1	42	28	42
-6	2	1	156	148	10
-5	2	1	148	147	7
-4	2	1	0	29	1
-3	2	1	68	66	12
-2	2	1	100	96	8
-1	2	1	153	155	5
0	2	1	432	407	8
1	2	1	396	375	5
2	2	1	637	661	12
3	2	1	197	205	4
4	2	1	71	75	9
5	2	1	37	42	37
6	2	1	0	9	1
7	2	1	178	158	8
8	2	1	42	68	42
9	2	1	190	182	7
10	2	1	40	56	40
11	2	1	53	40	35
-9	3	1	17	9	16
-8	3	1	95	83	19
-7	3	1	70	75	25
-6	3	1	110	120	15
-5	3	1	17	17	16
-4	3	1	97	69	9
-3	3	1	157	158	5
-2	3	1	13	11	13
-1	3	1	1247	1199	16
0	3	1	1313	1306	27
1	3	1	356	334	5
2	3	1	869	809	11
3	3	1	148	147	9
4	3	1	60	41	25
5	3	1	150	144	6
6	3	1	203	209	5
7	3	1	175	179	6
8	3	1	44	38	43
9	3	1	188	186	8
10	3	1	46	18	46
11	3	1	111	124	13
-9	4	1	45	36	45
-8	4	1	96	100	16
-7	4	1	34	26	33
-6	4	1	55	51	54
-5	4	1	151	153	10
-4	4	1	267	251	6
-3	4	1	559	551	8
-2	4	1	129	132	6
-1	4	1	308	292	5
0	4	1	164	169	7
1	4	1	600	585	8
2	4	1	228	215	7
3	4	1	53	58	37
4	4	1	16	11	15
5	4	1	258	268	8
6	4	1	233	235	9
7	4	1	202	180	10
8	4	1	62	65	18
9	4	1	0	27	1
10	4	1	28	8	28
11	4	1	39	36	38
-8	5	1	113	117	14
-7	5	1	26	34	26
-6	5	1	70	3	20
-5	5	1	83	63	27
-4	5	1	78	85	12
-3	5	1	214	194	8
-2	5	1	164	161	7
-1	5	1	246	229	7
0	5	1	35	33	35
1	5	1	253	54	81
2	5	1	589	563	9
3	5	1	259	251	6
4	5	1	220	190	10
5	5	1	139	133	13
6	5	1	199	178	11
7	5	1	79	80	29
8	5	1	153	131	16
9	5	1	46	6	45
10	5	1	0	2	1
-8	6	1	32	51	31
-7	6	1	77	43	23
-6	6	1	0	56	1
-5	6	1	0	4	1
-4	6	1	69	79	23
-3	6	1	79	90	15
-2	6	1	255	261	6
-1	6	1	98	118	11
0	6	1	454	430	7
1	6	1	68	69	20
2	6	1	52	35	35
3	6	1	40	50	40
4	6	1	136	121	10
5	6	1	6	55	6
6	6	1	117	113	17
7	6	1	54	30	53
8	6	1	68	37	67
9	6	1	214	226	14
10	6	1	70	39	70
-7	7	1	25	46	24
-6	7	1	0	39	1
-5	7	1	58	55	35
-4	7	1	114	107	16
-3	7	1	109	117	11
-2	7	1	238	235	7
-1	7	1	0	5	1
0	7	1	34	54	34
1	7	1	364	272	14
2	7	1	197	179	8
3	7	1	22	8	22
4	7	1	70	63	23
5	7	1	97	101	17
6	7	1	7	22	6
7	7	1	72	44	71
8	7	1	211	140	26
9	7	1	113	83	38
10	7	1	150	149	25
-6	8	1	28	14	27
-5	8	1	116	134	14
-4	8	1	21	44	21
-3	8	1	164	176	10
-2	8	1	63	74	30
-1	8	1	65	53	22
0	8	1	202	205	9
1	8	1	122	120	13
2	8	1	145	145	10
3	8	1	160	147	10
4	8	1	33	5	33
5	8	1	130	116	15
6	8	1	0	19	1
7	8	1	0	5	1
8	8	1	111	97	26
9	8	1	71	76	70
10	8	1	93	97	40
-6	9	1	55	95	54
-5	9	1	36	28	35
-4	9	1	46	49	46
-3	9	1	86	91	27
-2	9	1	138	126	14
-1	9	1	29	38	28
0	9	1	127	140	12
1	9	1	146	149	12
2	9	1	212	216	10
3	9	1	13	33	12
4	9	1	159	134	13
5	9	1	142	122	15
6	9	1	67	75	39
7	9	1	27	29	26
8	9	1	102	96	25
9	9	1	51	88	50
-5	10	1	37	35	36
-4	10	1	109	124	22
-3	10	1	209	191	14
-2	10	1	251	268	11
-1	10	1	221	236	8
0	10	1	53	51	53
1	10	1	0	34	1
2	10	1	10	15	10
3	10	1	74	71	38
4	10	1	46	41	45
5	10	1	121	114	19
6	10	1	315	326	8
7	10	1	0	5	1
8	10	1	64	60	63
9	10	1	46	45	46
-4	11	1	64	86	59
-3	11	1	70	86	69
-2	11	1	177	162	22
-1	11	1	170	170	11
0	11	1	0	74	1
1	11	1	30	52	29
2	11	1	81	38	35
3	11	1	0	20	1
4	11	1	51	48	51
5	11	1	0	40	1
6	11	1	326	346	9
7	11	1	0	10	1
8	11	1	0	23	1
-2	12	1	187	188	21
-1	12	1	31	53	30
0	12	1	197	193	10
1	12	1	68	83	39
2	12	1	38	1	37
3	12	1	78	74	31
4	12	1	53	22	53
5	12	1	28	53	27
6	12	1	109	111	21
7	12	1	0	16	1
0	13	1	73	84	49
1	13	1	166	120	20
2	13	1	140	124	17
3	13	1	22	17	22
4	13	1	118	129	19
5	13	1	71	56	42
-5	-12	2	83	26	26
-4	-12	2	0	9	1
-3	-12	2	53	97	53
-2	-12	2	26	48	26
-1	-12	2	37	6	36
0	-12	2	50	55	50
1	-12	2	172	172	15
-6	-11	2	54	10	54
-5	-11	2	213	192	12
-4	-11	2	0	59	1
-3	-11	2	0	29	1
-2	-11	2	130	124	16
-1	-11	2	65	76	65
0	-11	2	73	59	42
1	-11	2	158	118	17
2	-11	2	0	5	1
3	-11	2	82	26	44
-7	-10	2	120	129	18
-6	-10	2	39	38	38
-5	-10	2	230	205	12
-4	-10	2	97	119	29
-3	-10	2	87	100	26
-2	-10	2	43	10	42
-1	-10	2	85	85	26
0	-10	2	0	19	1
1	-10	2	195	207	13
2	-10	2	240	239	19
3	-10	2	80	92	27
4	-10	2	64	60	63
-8	-9	2	146	141	18
-7	-9	2	138	139	19
-6	-9	2	42	0	42
-5	-9	2	94	107	25
-4	-9	2	73	74	44
-3	-9	2	129	125	15
-2	-9	2	28	40	28
-1	-9	2	0	30	1
0	-9	2	258	240	8
1	-9	2	76	79	37
2	-9	2	174	175	14
3	-9	2	235	235	19
4	-9	2	257	242	13
5	-9	2	73	63	52
6	-9	2	0	8	1
-8	-8	2	0	33	1
-7	-8	2	119	136	26
-6	-8	2	112	102	28
-5	-8	2	168	162	18
-4	-8	2	0	31	1
-3	-8	2	0	6	1
-2	-8	2	72	77	21
-1	-8	2	69	65	27
0	-8	2	327	323	7
1	-8	2	224	220	8
2	-8	2	274	259	10
3	-8	2	227	236	10
4	-8	2	154	151	14
5	-8	2	116	105	25
6	-8	2	84	72	32
-9	-7	2	107	132	30
-8	-7	2	142	124	18
-7	-7	2	67	66	66
-6	-7	2	173	155	17
-5	-7	2	0	13	1
-4	-7	2	84	61	32
-3	-7	2	328	315	8
-2	-7	2	227	205	10
-1	-7	2	214	217	7
0	-7	2	248	256	7
1	-7	2	97	99	16
2	-7	2	60	46	32
3	-7	2	32	43	32
4	-7	2	124	121	17
5	-7	2	103	92	27
6	-7	2	56	27	56
7	-7	2	89	110	26
-9	-6	2	57	88	57
-8	-6	2	22	1	22
-7	-6	2	23	1	23
-6	-6	2	262	280	10
-5	-6	2	198	200	13
-4	-6	2	382	368	9
-3	-6	2	196	206	10
-2	-6	2	160	153	9
-1	-6	2	366	363	7
0	-6	2	659	620	9
1	-6	2	273	276	6
2	-6	2	18	0	17
3	-6	2	32	27	32
4	-6	2	116	103	10
5	-6	2	124	125	17
6	-6	2	36	25	35
7	-6	2	0	29	1
8	-6	2	83	104	27
-9	-5	2	92	55	44
-8	-5	2	79	82	78
-7	-5	2	113	112	19
-6	-5	2	123	101	18
-5	-5	2	200	208	9
-4	-5	2	60	37	59
-3	-5	2	94	84	22
-2	-5	2	418	425	8
-1	-5	2	174	178	7
0	-5	2	15	16	15
1	-5	2	274	287	5
2	-5	2	0	10	1
3	-5	2	122	118	8
4	-5	2	173	178	7
5	-5	2	58	28	27
6	-5	2	69	34	51
7	-5	2	60	72	60
8	-5	2	43	66	42
9	-5	2	34	55	33
-10	-4	2	104	114	33
-9	-4	2	51	38	51
-8	-4	2	76	93	35
-7	-4	2	166	144	12
-6	-4	2	7	39	6
-5	-4	2	201	200	9
-4	-4	2	267	248	10
-3	-4	2	124	97	15
-2	-4	2	74	74	22
-1	-4	2	52	49	42
0	-4	2	76	64	15
1	-4	2	219	219	6
2	-4	2	276	280	5
3	-4	2	45	49	26
4	-4	2	200	187	9
5	-4	2	146	144	8
6	-4	2	141	124	10
7	-4	2	46	35	46
8	-4	2	64	92	63
9	-4	2	87	95	25
-10	-3	2	65	75	43
-9	-3	2	153	147	15
-8	-3	2	73	82	34
-7	-3	2	70	78	28
-6	-3	2	53	80	26
-5	-3	2	23	1	23
-4	-3	2	85	79	20
-3	-3	2	131	121	9
-2	-3	2	176	175	7
-1	-3	2	110	111	14
0	-3	2	839	791	13
1	-3	2	363	382	6
2	-3	2	0	16	1
3	-3	2	242	141	49
4	-3	2	125	117	10
5	-3	2	26	26	26
6	-3	2	62	57	21
7	-3	2	0	0	1
8	-3	2	48	10	47
9	-3	2	0	10	1
-10	-2	2	43	32	42
-9	-2	2	290	280	7
-8	-2	2	213	207	7
-7	-2	2	42	10	41
-6	-2	2	83	82	10
-5	-2	2	141	141	8
-4	-2	2	510	532	9
-3	-2	2	394	376	6
-2	-2	2	992	986	13
-1	-2	2	1107	1123	20
0	-2	2	702	694	19
1	-2	2	59	38	16
2	-2	2	216	186	6
3	-2	2	97	92	9
4	-2	2	137	125	7
5	-2	2	315	321	6
6	-2	2	57	52	23
7	-2	2	98	113	11
8	-2	2	42	54	42
9	-2	2	18	15	18
10	-2	2	155	161	15
-10	-1	2	171	180	8
-9	-1	2	133	138	10
-8	-1	2	72	65	19
-7	-1	2	141	146	9
-6	-1	2	36	31	35
-5	-1	2	0	19	1
-4	-1	2	131	141	5
-3	-1	2	132	40	48
-2	-1	2	128	113	8
-1	-1	2	304	296	5
0	-1	2	136	148	6
1	-1	2	23	25	22
2	-1	2	411	403	6
3	-1	2	106	107	8
4	-1	2	38	31	37
5	-1	2	135	133	7
6	-1	2	347	344	6
7	-1	2	187	198	6
8	-1	2	49	47	33
9	-1	2	0	41	1
10	-1	2	103	98	15
-9	0	2	47	41	46
-8	0	2	160	153	9
-7	0	2	151	142	7
-6	0	2	316	331	7
-5	0	2	108	103	12
-4	0	2	33	21	32
-3	0	2	198	198	4
-2	0	2	95	92	7
-1	0	2	1508	1535	20
0	0	2	316	302	4
1	0	2	245	255	5
2	0	2	1226	1251	18
3	0	2	139	129	10
4	0	2	273	296	6

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
5	0	2	311	365	20
6	0	2	165	183	8
7	0	2	199	207	7
8	0	2	71	58	23
9	0	2	0	46	1
10	0	2	229	212	9
-9	1	2	33	45	32
-8	1	2	119	133	14
-7	1	2	93	114	21
-6	1	2	149	152	12
-5	1	2	0	34	1
-4	1	2	0	19	1
-3	1	2	298	237	21
-2	1	2	527	502	8
-1	1	2	278	268	5
0	1	2	106	159	13
1	1	2	300	308	8
2	1	2	278	295	5
3	1	2	0	76	1
4	1	2	60	90	26
5	1	2	288	286	8
6	1	2	75	60	18
7	1	2	263	250	7
8	1	2	84	97	19
9	1	2	370	372	11
10	1	2	0	3	1
11	1	2	101	103	19
-9	2	2	201	205	9
-8	2	2	148	160	13
-7	2	2	81	59	26
-6	2	2	285	292	8
-5	2	2	181	179	9
-4	2	2	405	421	8
-3	2	2	82	94	8
-2	2	2	33	21	33
-1	2	2	274	265	6
0	2	2	82	92	7
1	2	2	1859	1662	45
2	2	2	573	611	6
3	2	2	481	534	9
5	2	2	58	43	28
6	2	2	40	28	40
7	2	2	205	198	9
8	2	2	70	61	35
9	2	2	63	85	41
10	2	2	20	13	19
11	2	2	77	85	17
-9	3	2	39	28	38
-8	3	2	65	39	32
-7	3	2	79	74	23
-6	3	2	215	226	8
-5	3	2	92	77	19
-4	3	2	0	12	1
-3	3	2	41	33	41
-2	3	2	53	34	29
-1	3	2	70	63	14
0	3	2	526	520	8
1	3	2	33	6	32
2	3	2	847	792	12
3	3	2	591	608	9
4	3	2	981	1060	12
5	3	2	315	313	5
6	3	2	25	4	24
7	3	2	17	22	17
8	3	2	122	98	10
9	3	2	110	110	13
10	3	2	226	215	7
11	3	2	138	150	11
-8	4	2	115	129	15
-7	4	2	93	74	21
-6	4	2	50	46	49
-5	4	2	38	31	38
-4	4	2	273	286	7
-3	4	2	250	245	5
-2	4	2	248	140	51
-1	4	2	514	504	7
0	4	2	191	201	7
1	4	2	732	728	10
2	4	2	809	799	10
3	4	2	889	821	13
4	4	2	156	157	7
5	4	2	316	326	8
6	4	2	222	225	5
7	4	2	111	111	10
8	4	2	181	177	7
9	4	2	70	66	18
10	4	2	95	75	14
11	4	2	39	76	39
-8	5	2	37	59	37
-7	5	2	118	124	16
-6	5	2	0	20	1
-5	5	2	70	51	22
-4	5	2	138	148	10
-3	5	2	338	329	6
-2	5	2	223	144	15
-1	5	2	205	187	7
0	5	2	122	118	11
1	5	2	461	454	7
2	5	2	378	377	6
3	5	2	138	137	8
4	5	2	91	71	16
5	5	2	379	356	8
6	5	2	202	139	18
7	5	2	0	41	1
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8	6	2	66	43	65
9	6	2	151	135	20
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8	0	3	37	56	36
9	0	3	0	10	1
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Table 6. Observed and calculated structure factors for af011

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6	2	3	283	306	8
7	2	3	230	238	8
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9	2	3	136	159	15
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2	3	3	18	4	17
3	3	3	628	611	9
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10	4	3	168	160	8
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8	6	3	163	125	17
9	6	3	153	152	17
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3	0	4	254	282	7
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6	0	4	49	74	31
7	0	4	113	95	11
8	0	4	36	42	36
9	0	4	89	87	18
10	0	4	136	148	14
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3	1	4	795	835	10
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8	1	4	122	100	16
9	1	4	63	85	30
10	1	4	64	54	27
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Table 6. Observed and calculated structure factors for af011

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4	3	4	296	304	5
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6	3	4	130	119	8
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8	3	4	0	99	1
9	3	4	96	100	21
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3	4	4	364	379	11
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7	4	4	362	343	5
8	4	4	195	189	6
9	4	4	112	96	11
10	4	4	260	251	7
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7	7	4	63	40	43
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9	7	4	77	58	70
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-6	3	5	14	10	13
-5	3	5	71	58	29
-4	3	5	109	81	14
-3	3	5	146	142	6
-2	3	5	12	7	12
-1	3	5	52	57	11
0	3	5	380	379	4
1	3	5	437	465	23
2	3	5	353	354	6
3	3	5	60	50	12
4	3	5	128	123	5
5	3	5	118	105	7
6	3	5	386	378	7
7	3	5	167	174	7
8	3	5	153	153	9
9	3	5	255	277	10
10	3	5	0	56	1
11	3	5	13	48	13
-7	4	5	207	219	18
-6	4	5	292	304	8
-5	4	5	0	14	1
-4	4	5	22	18	21
-3	4	5	25	3	25
-2	4	5	244	242	5
-1	4	5	0	16	1
0	4	5	34	11	33
1	4	5	40	54	30
2	4	5	220	229	4
3	4	5	566	570	15

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
4	4	5	12	29	12
5	4	5	51	69	50
6	4	5	479	500	8
7	4	5	117	111	9
8	4	5	57	45	28
9	4	5	83	100	17
10	4	5	155	157	11
-7	5	5	65	32	36
-6	5	5	103	109	20
-5	5	5	87	104	22
-4	5	5	184	192	10
-3	5	5	68	58	30
-2	5	5	0	2	1
-1	5	5	162	159	9
0	5	5	292	292	7
1	5	5	111	114	11
2	5	5	338	321	6
3	5	5	178	162	14
4	5	5	372	378	12
5	5	5	217	197	6
6	5	5	0	5	1
7	5	5	157	151	5
8	5	5	75	75	11
9	5	5	81	74	12
10	5	5	138	122	22
11	5	5	49	12	49
-7	6	5	94	44	19
-6	6	5	135	127	13
-5	6	5	84	77	25
-4	6	5	204	222	9
-3	6	5	127	138	14
-2	6	5	134	117	12
-1	6	5	0	23	1
0	6	5	268	271	7
1	6	5	203	181	7
2	6	5	42	45	42
3	6	5	514	490	8
4	6	5	101	96	6
5	6	5	92	88	14
6	6	5	199	179	11
7	6	5	62	36	51
8	6	5	62	45	62
9	6	5	60	19	59
10	6	5	60	17	60
11	6	5	156	153	22
-6	7	5	39	34	38
-5	7	5	0	9	1
-4	7	5	64	30	42
-3	7	5	166	173	10
-2	7	5	97	101	20
-1	7	5	180	188	8
0	7	5	543	522	9
1	7	5	172	165	8
2	7	5	93	53	15
3	7	5	116	93	9
4	7	5	193	195	6
5	7	5	380	365	7
6	7	5	157	147	11
7	7	5	87	85	46
8	7	5	39	47	38
9	7	5	129	122	36
10	7	5	0	31	1
11	7	5	49	32	49
-5	8	5	41	56	41
-4	8	5	60	76	41
-3	8	5	117	115	18
-2	8	5	57	62	48
-1	8	5	166	170	10
0	8	5	197	192	9
1	8	5	114	105	15
2	8	5	59	60	27
3	8	5	68	84	17
4	8	5	126	131	9
5	8	5	228	229	15
6	8	5	35	9	35
7	8	5	82	72	82
8	8	5	56	19	55
9	8	5	0	24	1
10	8	5	35	19	34
11	8	5	33	87	33
-5	9	5	0	28	1
-4	9	5	66	52	36
-3	9	5	45	87	44
-2	9	5	141	129	16
-1	9	5	51	71	51
0	9	5	25	9	24
1	9	5	145	137	12
2	9	5	120	129	14
3	9	5	168	170	8
4	9	5	190	154	28
5	9	5	56	2	55
6	9	5	209	213	18
7	9	5	0	87	1
8	9	5	181	187	22
9	9	5	89	86	70
10	9	5	0	36	1
-4	10	5	0	11	1
-3	10	5	46	92	46
-2	10	5	168	162	12
-1	10	5	103	67	21
0	10	5	6	7	6
1	10	5	275	272	9
2	10	5	146	131	15
3	10	5	0	22	1
4	10	5	113	120	39
5	10	5	70	73	70
6	10	5	279	279	17
7	10	5	118	107	43
8	10	5	41	81	41
9	10	5	127	111	32
10	10	5	81	67	80
-3	11	5	7	5	7
-2	11	5	20	45	20
-1	11	5	160	160	12
0	11	5	51	59	50
1	11	5	36	45	35
2	11	5	72	85	72
3	11	5	167	142	31
4	11	5	77	103	77
5	11	5	60	62	59
6	11	5	80	55	80
7	11	5	262	264	18
8	11	5	39	54	39
9	11	5	196	182	23
-2	12	5	0	21	1
-1	12	5	0	19	1
0	12	5	182	181	11
1	12	5	45	35	44
2	12	5	443	454	19
3	12	5	132	137	40
4	12	5	105	118	39
5	12	5	5	5	5
6	12	5	73	62	73
7	12	5	0	29	1
8	12	5	220	216	21
0	13	5	89	108	73
1	13	5	51	32	51
2	13	5	277	274	23
3	13	5	47	73	46
4	13	5	0	47	1
5	13	5	73	53	73
6	13	5	0	33	1
7	13	5	90	119	51
3	14	5	47	12	46
4	14	5	0	43	1
5	14	5	94	104	58
-3	-10	6	26	20	25
-2	-10	6	139	131	18
-1	-10	6	271	288	11
0	-10	6	130	124	19
1	-10	6	9	39	8
2	-10	6	50	28	50
-4	-9	6	104	101	27
-3	-9	6	66	76	47
-2	-9	6	0	13	1
-1	-9	6	0	16	1
0	-9	6	69	62	61
1	-9	6	0	3	1
2	-9	6	88	96	32
3	-9	6	122	109	20
4	-9	6	150	160	17
-5	-8	6	29	28	28
-4	-8	6	0	41	1
-3	-8	6	261	255	12
-2	-8	6	169	180	15
-1	-8	6	0	14	1
0	-8	6	0	11	1
1	-8	6	122	125	23
2	-8	6	0	5	1
3	-8	6	94	104	30
4	-8	6	174	171	16
5	-8	6	80	27	33
-6	-7	6	0	60	1
-5	-7	6	47	53	47
-4	-7	6	120	127	19
-3	-7	6	82	72	34
-2	-7	6	0	18	1
-1	-7	6	27	29	26
0	-7	6	120	126	21
1	-7	6	31	8	30
2	-7	6	91	84	35
3	-7	6	173	173	17
4	-7	6	334	329	11
5	-7	6	0	25	1
6	-7	6	158	143	16
-7	-6	6	181	154	11
-6	-6	6	0	39	1
-5	-6	6	83	87	30
-4	-6	6	210	209	12
-3	-6	6	282	308	9
-2	-6	6	0	19	1
-1	-6	6	259	259	11
0	-6	6	119	104	21
1	-6	6	45	29	44
2	-6	6	48	50	48
3	-6	6	168	172	15
4	-6	6	167	163	17
5	-6	6	349	349	10
6	-6	6	36	6	35
7	-6	6	78	76	32
-7	-5	6	70	30	46
-6	-5	6	0	4	1
-5	-5	6	45	35	44
-4	-5	6	39	48	39
-3	-5	6	215	217	9
-2	-5	6	261	270	8
-1	-5	6	198	204	10
0	-5	6	207	212	11
1	-5	6	158	152	13
2	-5	6	99	104	22
3	-5	6	159	151	15
4	-5	6	99	93	24
5	-5	6	252	250	10
6	-5	6	26	47	26
7	-5	6	0	17	1
8	-5	6	35	37	35
-7	-4	6	62	22	61
-6	-4	6	0	52	1
-5	-4	6	66	60	35
-4	-4	6	33	14	33
-3	-4	6	0	14	1
-2	-4	6	100	108	15
-1	-4	6	178	163	8
0	-4	6	68	81	26
1	-4	6	214	218	9
2	-4	6	85	86	13
3	-4	6	57	38	57
4	-4	6	122	132	18
5	-4	6	119	114	17
6	-4	6	213	205	10
7	-4	6	291	264	10
8	-4	6	156	146	12
9	-4	6	116	137	19
-8	-3	6	38	6	37
-7	-3	6	63	43	63
-6	-3	6	0	8	1
-5	-3	6	27	15	27
-4	-3	6	70	61	25
-3	-3	6	25	40	24
-2	-3	6	52	51	44
-1	-3	6	33	8	33
0	-3	6	145	130	10
1	-3	6	113	113	11
2	-3	6	118	139	15
3	-3	6	255	256	9
4	-3	6	223	204	9
5	-3	6	90	95	21
6	-3	6	63	64	40
7	-3	6	0	3	1
8	-3	6	240	233	9
9	-3	6	0	12	1
-8	-2	6	42	0	41
-7	-2	6	102	99	23
-6	-2	6	58	18	43
-5	-2	6	18	3	17
-4	-2	6	81	71	15
-3	-2	6	247	235	7
-2	-2	6	256	238	7
-1	-2	6	103	118	14
0	-2	6	93	78	15
1	-2	6	237	222	6
2	-2	6	127	131	9
3	-2	6	322	312	8
4	-2	6	206	186	9
5	-2	6	111	113	15
6	-2	6	89	68	20
7	-2	6	40	16	40
8	-2	6	154	153	13
9	-2	6	44	23	43
10	-2	6	11	44	11
-8	-1	6	0	7	1
-7	-1	6	45	10	44
-6	-1	6	0	32	1
-5	-1	6	0	23	1
-4	-1	6	93	97	15
-3	-1	6	90	83	15
-2	-1	6	0	25	1
-1	-1	6	197	182	8
0	-1	6	124	124	11
1	-1	6	67	34	25
2	-1	6	232	236	5
3	-1	6	583	565	8
4	-1	6	12	8	11
5	-1	6	76	71	14
6	-1	6	51	15	51
7	-1	6	52	41	52
8	-1	6	0	14	1
9	-1	6	122	132	14
10	-1	6	32	43	32
-8	0	6	37	60	36
-7	0	6	63	64	63
-6	0	6	47	42	46
-5	0	6	68	52	30
-4	0	6	43	13	43
-3	0	6	111	102	11
-2	0	6	314	327	6
-1	0	6	298	263	16
0	0	6	202	200	6
1	0	6	305	311	5
2	0	6	115	110	8
3	0	6	191	189	9
4	0	6	190	201	9
5	0	6	34	34	34
6	0	6	106	112	10
7	0	6	200	183	9
8	0	6	21	56	21
9	0	6	0	20	1
10	0	6	4	28	4
-8	1	6	50	34	50
-7	1	6	113	105	13
-6	1	6	56	41	37
-5	1	6	142	153	9
-4	1	6	58	67	22
-3	1	6	95	79	11
-2	1	6	207	208	6
-1	1	6	29	24	29
0	1	6	207	215	7
1	1	6	258	290	13
2	1	6	236	221	6
3	1	6	576	594	8
4	1	6	202	199	5
5	1	6	260	255	5
6	1	6	139	134	7
7	1	6	0	4	1
8	1	6	0	20	1
9	1	6	0	15	1
10	1	6	124	127	13
11	1	6	149	159	10
-7	2	6	20	9	20
-6	2	6	70	72	22
-5	2	6	0	15	1
-4	2	6	32	23	31
-3	2	6	117	119	7
-2	2	6	117	108	6
-1	2	6	492	498	7
0	2	6	0	1	1
1	2	6	224	223	6
2	2	6	70	79	15
3	2	6	29	38	29
4	2	6	96	89	9
5	2	6	450	436	6
6	2	6	267	271	5
7	2	6	73	63	16
8	2	6	78	92	15
9	2	6	97	89	14
10	2	6	40	59	40
11	2	6	70	83	21
-7	3	6	10	3	10
-6	3	6	86	74	17
-5	3	6	28	35	28
-4	3	6	199	182	7
-3	3	6	221	218	5
-2	3	6	34	14	33
-1	3	6	162	162	4
0	3	6	164	153	5
1	3	6	53	66	14
2	3	6	76	61	12
3	3	6	52	22	18
4	3	6	372	371	6
5	3	6	0	18	1
6	3	6	241	256	6
7	3	6	39	35	39
8	3	6	55	59	33
9	3	6	84	75	19
10	3	6	0	50	1
11	3	6	0	75	1
-7	4	6	173	165	10
-6	4	6	128	116	9
-5	4	6	325	338	11
-4	4	6	89	104	19
-3	4	6	42	48	42
-2	4	6	157	147	6
-1	4	6	202	201	4
0	4	6	299	297	4
1	4	6	218	213	3
2	4	6	204	212	6
3	4	6	192	175	13
4	4	6	120	111	4
5	4	6	90	98	7
6	4	6	344	349	7
7	4	6	292	284	7
8	4	6	21	14	20
9	4	6	212	191	8
10	4	6	102	119	15
11	4	6	61	76	60
-6	5	6	47	41	46
-5	5	6	157	181	14
-4	5	6	63	55	37
-3	5	6	0	14	1
-2	5	6	43	47	43
-1	5	6	86	77	11
0	5	6	188	182	6
1	5	6	264	264	6
2	5	6	401	407	4
3	5	6	59	71	58
4	5	6	110	78	32
5	5	6	100	93	10
6	5	6	109	104	7
7	5	6	77	64	10
8	5	6	98	90	28
9	5	6	40	19	40
10	5	6	83	85	50
11	5	6	142	163	23
-6	6	6	124	135	16
-5	6	6	0	46	1
-4	6	6	39	14	38
-3	6	6	98	87	20
-2	6	6	184	203	8
-1	6	6	8	15	8
0	6	6	138	137	10
1	6	6	178	174	8
2	6	6	409	406	7
3	6	6	233	243	13
4	6	6	325	321	12
5	6	6	56	65	55
6	6	6	254	244	14
7	6	6	207	168	16
8	6	6	266	259	16
9	6	6	89	85	57
10	6	6	87	82	75
11	6	6	134	137	27
-6	7	6	0	85	1
-5	7	6	38	48	38
-4	7	6	237	231	9
-3	7	6	82	73	24
-2	7	6	137	123	13
-1	7	6	0	26	1
0	7	6	383	378	8
1	7	6	328	324	7
2	7	6	298	309	6
3	7	6	384	362	7
4	7	6	303	299	13
5	7	6	207	221	13
6	7	6	110	121	30
7	7	6	23	53	23

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
8	7	6	53	28	53
9	7	6	69	45	69
10	7	6	49	12	48
11	7	6	0	20	1
-5	8	6	113	112	16
-4	8	6	61	52	36
-3	8	6	114	90	18
-2	8	6	80	93	28
-1	8	6	147	141	14
0	8	6	150	148	10
1	8	6	75	62	25
2	8	6	181	169	8
3	8	6	41	35	41
4	8	6	51	76	51
5	8	6	261	277	14
6	8	6	202	196	17
7	8	6	220	249	16
8	8	6	180	147	24
9	8	6	0	20	1
10	8	6	0	1	1
11	8	6	54	75	53
-4	9	6	0	30	1
-3	9	6	22	15	21
-2	9	6	229	228	10
-1	9	6	228	243	9
0	9	6	117	117	16
1	9	6	58	60	57
2	9	6	76	38	27
3	9	6	165	163	24
4	9	6	62	61	61
5	9	6	140	144	25
6	9	6	26	24	26
7	9	6	63	81	63
8	9	6	0	30	1
9	9	6	56	49	55
10	9	6	94	80	50
-3	10	6	139	116	14
-2	10	6	229	230	9
-1	10	6	161	149	14
0	10	6	216	212	11
1	10	6	96	75	21
2	10	6	96	63	96
3	10	6	141	112	35
4	10	6	0	33	1
5	10	6	44	41	43
6	10	6	0	39	1
7	10	6	195	240	21
8	10	6	0	55	1
9	10	6	158	162	29
10	10	6	54	92	53
-2	11	6	0	5	1
-1	11	6	217	219	9
0	11	6	37	1	36
1	11	6	0	5	1
2	11	6	0	13	1
3	11	6	42	6	41
4	11	6	186	144	28
5	11	6	57	86	57
6	11	6	134	143	34
7	11	6	0	16	1
8	11	6	26	17	25
9	11	6	32	26	32
-1	12	6	54	38	53
0	12	6	86	75	85
1	12	6	16	29	16
2	12	6	155	170	35
3	12	6	61	57	61
4	12	6	67	98	66
5	12	6	150	142	34
6	12	6	124	149	39
7	12	6	16	21	16
8	12	6	70	91	69
0	13	6	62	46	62
1	13	6	0	25	1
2	13	6	76	79	76
3	13	6	181	178	31
4	13	6	0	40	1
5	13	6	229	260	16
6	13	6	0	1	1
7	13	6	0	71	1
3	14	6	124	118	61
4	14	6	27	33	27
5	14	6	45	23	44
-3	-9	7	129	131	19
-2	-9	7	16	0	16
-1	-9	7	0	8	1
0	-9	7	78	81	36
1	-9	7	136	143	20
2	-9	7	0	0	1
3	-9	7	0	2	1
-4	-8	7	11	33	10
-3	-8	7	198	212	13
-2	-8	7	0	13	1
-1	-8	7	74	71	41
0	-8	7	0	34	1
1	-8	7	16	0	16
2	-8	7	0	19	1
3	-8	7	102	98	26
4	-8	7	82	99	39
5	-8	7	60	22	59
-5	-7	7	57	46	57
-4	-7	7	90	83	25
-3	-7	7	54	53	53
-2	-7	7	67	50	67
-1	-7	7	118	126	23
0	-7	7	85	96	32
1	-7	7	49	56	48
2	-7	7	79	70	44
3	-7	7	0	25	1
4	-7	7	266	252	11
5	-7	7	258	254	11
6	-7	7	24	13	23
-6	-6	7	0	6	1
-5	-6	7	201	192	11
-4	-6	7	257	252	9
-3	-6	7	213	224	11
-2	-6	7	107	114	25
-1	-6	7	180	176	12
0	-6	7	124	111	21
1	-6	7	23	34	22
2	-6	7	46	42	45
3	-6	7	172	154	18
4	-6	7	25	7	24
5	-6	7	59	69	58
6	-6	7	243	236	10
7	-6	7	58	33	57
-6	-5	7	44	29	43
-5	-5	7	100	111	21
-4	-5	7	63	72	36
-3	-5	7	133	133	14
-2	-5	7	36	35	36
-1	-5	7	200	197	9
0	-5	7	222	219	11
1	-5	7	36	31	35
2	-5	7	0	11	1
3	-5	7	65	66	65
4	-5	7	52	43	51
5	-5	7	91	88	28
6	-5	7	220	212	11
7	-5	7	61	46	60
8	-5	7	89	81	28
-7	-4	7	47	60	46
-6	-4	7	109	140	21
-5	-4	7	11	26	10
-4	-4	7	107	94	17
-3	-4	7	107	118	16
-2	-4	7	135	114	14
-1	-4	7	0	24	1
0	-4	7	267	248	8
1	-4	7	129	127	16
2	-4	7	248	236	10
3	-4	7	168	135	14
4	-4	7	169	173	13
5	-4	7	72	34	38
6	-4	7	71	83	35
7	-4	7	67	82	67
8	-4	7	148	127	15
-7	-3	7	0	58	1
-6	-3	7	48	77	48
-5	-3	7	0	12	1
-4	-3	7	37	53	36
-3	-3	7	24	30	24
-2	-3	7	55	46	46
-1	-3	7	0	21	1
0	-3	7	32	13	31
1	-3	7	240	235	6
2	-3	7	146	125	14
3	-3	7	109	103	19
4	-3	7	198	179	11
5	-3	7	152	159	14
6	-3	7	60	55	59
7	-3	7	163	152	17
8	-3	7	213	214	11
9	-3	7	0	29	1
-7	-2	7	50	69	50
-6	-2	7	72	66	46
-5	-2	7	29	42	28
-4	-2	7	73	79	17
-3	-2	7	25	17	24
-2	-2	7	0	19	1
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1	-2	7	105	107	16
2	-2	7	0	13	1
3	-2	7	102	81	17
4	-2	7	184	176	9
5	-2	7	64	64	42
6	-2	7	130	135	13
7	-2	7	173	175	11
8	-2	7	72	47	28
9	-2	7	47	34	46
10	-2	7	73	92	25
-7	-1	7	95	68	25
-6	-1	7	0	23	1
-5	-1	7	29	6	29
-4	-1	7	70	72	25
-3	-1	7	165	165	9
-2	-1	7	108	99	13
-1	-1	7	400	393	7
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1	-1	7	277	215	17
2	-1	7	379	382	6
3	-1	7	50	47	31
4	-1	7	28	36	27
5	-1	7	230	234	8
6	-1	7	58	59	42
7	-1	7	284	282	8
8	-1	7	151	140	13
9	-1	7	97	101	18
10	-1	7	121	129	14
-7	0	7	12	5	12
-6	0	7	70	79	43
-5	0	7	50	42	50
-4	0	7	123	113	15
-3	0	7	207	201	8
-2	0	7	313	316	7
-1	0	7	316	333	8
0	0	7	368	358	6
1	0	7	28	13	27
2	0	7	475	476	7
3	0	7	342	352	18
4	0	7	232	232	10
5	0	7	38	43	31
6	0	7	50	62	25
7	0	7	111	108	14
8	0	7	114	90	15
9	0	7	0	37	1
10	0	7	63	56	20
-7	1	7	91	72	28
-6	1	7	0	5	1
-5	1	7	216	195	10
-4	1	7	95	102	21
-3	1	7	90	87	14
-2	1	7	417	401	14
-1	1	7	258	262	6
0	1	7	288	288	6
1	1	7	52	63	26
2	1	7	358	350	6
3	1	7	336	334	6
4	1	7	250	242	5
5	1	7	164	164	7
6	1	7	106	83	9
7	1	7	128	103	9
8	1	7	50	81	34
9	1	7	34	65	33
10	1	7	40	50	40
11	1	7	44	29	44
-7	2	7	0	38	1
-6	2	7	176	161	9
-5	2	7	137	140	11
-4	2	7	107	130	11
-3	2	7	221	202	6
-2	2	7	359	370	6
-1	2	7	264	269	6
0	2	7	192	192	7
1	2	7	200	204	6
2	2	7	232	236	5
3	2	7	21	3	20
4	2	7	70	61	14
5	2	7	437	442	6
6	2	7	32	32	32
7	2	7	58	50	22
8	2	7	89	103	14
9	2	7	92	96	17
10	2	7	62	65	27
11	2	7	0	65	1
-7	3	7	58	71	31
-6	3	7	77	35	20
-5	3	7	229	216	7
-4	3	7	94	84	14
-3	3	7	86	78	13
-2	3	7	99	107	9
-1	3	7	181	158	6
0	3	7	261	251	5
1	3	7	251	256	11
2	3	7	49	65	28
3	3	7	319	325	5
4	3	7	281	271	5
5	3	7	183	175	6
6	3	7	256	252	6
7	3	7	108	124	8
8	3	7	181	173	8
9	3	7	178	169	9
10	3	7	86	96	21
11	3	7	14	6	14
-6	4	7	215	193	7
-5	4	7	234	229	8
-4	4	7	10	20	10
-3	4	7	17	31	16
-2	4	7	60	53	19
-1	4	7	298	307	4
0	4	7	243	246	4
1	4	7	107	101	8
2	4	7	153	142	6
3	4	7	130	112	7
4	4	7	42	35	22
5	4	7	232	221	5
6	4	7	119	110	7
7	4	7	148	151	8
8	4	7	127	135	11
9	4	7	48	53	47
10	4	7	332	315	15
11	4	7	36	26	36
-6	5	7	20	11	19
-5	5	7	76	76	16
-4	5	7	61	66	43
-3	5	7	0	29	1
-2	5	7	222	217	8
-1	5	7	127	140	10
0	5	7	89	101	8
1	5	7	375	380	4
2	5	7	121	126	8
3	5	7	410	414	7
4	5	7	0	19	1
5	5	7	152	95	28
6	5	7	127	125	19
7	5	7	110	89	29
8	5	7	55	72	54
9	5	7	63	61	63
10	5	7	232	238	16
11	5	7	64	58	63
-6	6	7	129	145	16
-5	6	7	0	27	1
-4	6	7	162	139	14
-3	6	7	126	136	17
-2	6	7	29	29	28
-1	6	7	238	221	8
0	6	7	164	172	7
1	6	7	344	347	7
2	6	7	250	250	4
3	6	7	253	255	5
4	6	7	158	156	18
5	6	7	62	68	61
6	6	7	49	71	48
7	6	7	56	83	56
8	6	7	66	77	65
9	6	7	133	124	27
10	6	7	42	5	41
11	6	7	76	91	75
-5	7	7	25	26	24
-4	7	7	166	154	12
-3	7	7	299	290	10
-2	7	7	103	93	19
-1	7	7	198	184	10
0	7	7	280	271	8
1	7	7	415	402	8
2	7	7	45	65	45
3	7	7	175	158	17
4	7	7	246	233	15
5	7	7	240	246	14
6	7	7	73	93	57
7	7	7	199	213	16
8	7	7	45	67	44
9	7	7	129	85	35
10	7	7	107	93	38
11	7	7	0	31	1
-4	8	7	39	15	39
-3	8	7	129	132	15
-2	8	7	117	114	19
-1	8	7	0	11	1
0	8	7	79	81	26
1	8	7	126	101	16
2	8	7	21	4	20
3	8	7	0	2	1
4	8	7	166	172	23
5	8	7	117	117	31
6	8	7	165	161	20
7	8	7	0	15	1
8	8	7	57	58	56
9	8	7	89	90	57
10	8	7	168	166	21
11	8	7	14	49	13
-4	9	7	126	112	15
-3	9	7	49	47	49
-2	9	7	83	103	25
-1	9	7	261	234	10
0	9	7	56	46	56
1	9	7	0	19	1
2	9	7	0	2	1
3	9	7	187	167	23
4	9	7	206	208	19
5	9	7	84	72	67
6	9	7	112	107	35
7	9	7	128	139	29
8	9	7	56	60	55
9	9	7	0	27	1
10	9	7	91	101	52
-3	10	7	23	72	22
-2	10	7	69	62	34
-1	10	7	96	72	25
0	10	7	379	379	9
1	10	7	0	13	1
2	10	7	115	109	49
3	10	7	141	111	40
4	10	7	88	84	87
5	10	7	35	29	35
6	10	7	235	236	21
7	10	7	64	73	63
8	10	7	0	18	1
9	10	7	81	98	80
10	10	7	70	80	70
-2	11	7	61	30	38
-1	11	7	0	16	1
0	11	7	76	70	76
1	11	7	316	343	17
2	11	7	26	40	26
3	11	7	49	12	48
4	11	7	222	219	22
5	11	7	47	55	47
6	11	7	11	24	10
7	11	7	211	213	20
8	11	7	234	259	18
9	11	7	210	210	21
-1	12	7	130	123	53
0	12	7	151	131	29
1	12	7	29	15	28
2	12	7	144	152	35
3	12	7	173	151	37
4	12	7	53	13	53
5	12	7	0	16	1
6	12	7	59	1	59
7	12	7	117	139	38
8	12	7	171	197	20
1	13	7	51	40	50
2	13	7	43	56	42
3	13	7	0	57	1
4	13	7	124	111	40
5	13	7	31	54	31
6	13	7	31	68	31
7	13	7	56	90	56
-3	-8	8	21	10	20
-2	-8	8	45	51	44
-1	-8	8	68	78	48
0	-8	8	129	109	20
1	-8	8	71	90	50

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
2	-8	8	20	14	19
3	-8	8	42	64	42
4	-8	8	62	40	62
-4	-7	8	103	115	20
-3	-7	8	219	237	11
-2	-7	8	111	103	25
-1	-7	8	32	43	32
0	-7	8	0	56	1
1	-7	8	7	36	6
2	-7	8	100	93	27
3	-7	8	206	179	14
4	-7	8	168	164	16
5	-7	8	90	90	29
-5	-6	8	53	81	53
-4	-6	8	30	27	29
-3	-6	8	133	139	16
-2	-6	8	250	272	11
-1	-6	8	249	252	10
0	-6	8	269	258	19
1	-6	8	76	95	45
2	-6	8	67	70	67
3	-6	8	99	98	26
4	-6	8	51	35	50
5	-6	8	17	41	17
6	-6	8	0	24	1
-5	-5	8	118	103	15
-4	-5	8	92	84	20
-3	-5	8	94	99	22
-2	-5	8	99	110	19
-1	-5	8	55	57	54
0	-5	8	69	51	55
1	-5	8	103	113	24
2	-5	8	105	114	24
3	-5	8	80	52	36
4	-5	8	50	59	50
5	-5	8	108	94	28
6	-5	8	147	138	17
7	-5	8	0	1	1
-6	-4	8	3	19	3
-5	-4	8	112	85	16
-4	-4	8	107	101	18
-3	-4	8	31	59	31
-2	-4	8	265	259	8
-1	-4	8	203	206	9
0	-4	8	144	131	14
1	-4	8	60	41	60
2	-4	8	133	109	18
3	-4	8	21	53	21
4	-4	8	68	61	50
5	-4	8	185	187	14
6	-4	8	83	80	33
7	-4	8	81	89	27
8	-4	8	17	35	17
-6	-3	8	46	10	45
-5	-3	8	38	63	38
-4	-3	8	54	60	53
-3	-3	8	62	59	41
-2	-3	8	49	37	48
-1	-3	8	56	54	42
0	-3	8	109	96	14
1	-3	8	115	91	14
2	-3	8	54	42	53
3	-3	8	28	26	27
4	-3	8	287	292	10
5	-3	8	90	92	27
6	-3	8	134	123	17
7	-3	8	190	198	11
8	-3	8	140	118	15
9	-3	8	26	36	25
-6	-2	8	38	24	37
-5	-2	8	51	52	50
-4	-2	8	0	14	1
-3	-2	8	30	30	30
-2	-2	8	36	22	36
-1	-2	8	98	91	18
0	-2	8	83	68	19
1	-2	8	215	198	9
2	-2	8	84	78	18
3	-2	8	164	158	13
4	-2	8	220	198	10
5	-2	8	273	270	9
6	-2	8	82	83	31
7	-2	8	17	27	16
8	-2	8	51	55	50
9	-2	8	177	182	10
-6	-1	8	79	63	37
-5	-1	8	8	38	8

h	k	l	10Fo	10Fc	10s
-4	-1	8	13	11	12
-3	-1	8	31	41	31
-2	-1	8	108	102	14
-1	-1	8	403	394	7
0	-1	8	40	51	40
1	-1	8	235	246	8
2	-1	8	240	184	13
3	-1	8	0	26	1
4	-1	8	94	85	19
5	-1	8	122	106	15
6	-1	8	66	80	33
7	-1	8	0	19	1
8	-1	8	175	157	11
9	-1	8	73	43	29
10	-1	8	0	37	1
-6	0	8	120	90	27
-5	0	8	93	62	34
-4	0	8	151	130	18
-3	0	8	104	94	16
-2	0	8	0	9	1
-1	0	8	37	55	36
0	0	8	113	116	10
1	0	8	52	42	29
2	0	8	48	32	48
3	0	8	123	131	10
4	0	8	81	88	12
5	0	8	37	15	37
6	0	8	22	6	22
7	0	8	33	21	33
8	0	8	212	213	9
9	0	8	295	290	8
10	0	8	145	162	11
-6	1	8	0	22	1
-5	1	8	162	126	15
-4	1	8	44	52	43
-3	1	8	230	236	8
-2	1	8	98	106	14
-1	1	8	78	56	18
0	1	8	138	133	9
1	1	8	127	136	10
2	1	8	175	178	7
3	1	8	258	267	6
4	1	8	241	238	6
5	1	8	458	445	7
6	1	8	31	5	31
7	1	8	20	12	20
8	1	8	104	66	19
9	1	8	77	85	23
10	1	8	30	50	29
-6	2	8	119	124	21
-5	2	8	24	29	23
-4	2	8	0	19	1
-3	2	8	0	1	1
-2	2	8	78	84	17
-1	2	8	95	90	13
0	2	8	67	66	20
1	2	8	189	194	6
2	2	8	298	300	6
3	2	8	382	391	6
4	2	8	29	39	29
5	2	8	245	240	5
6	2	8	157	159	8
7	2	8	84	80	14
8	2	8	73	84	22
9	2	8	131	103	12
10	2	8	60	26	30
11	2	8	68	54	68
-6	3	8	126	115	13
-5	3	8	11	5	10
-4	3	8	52	63	38
-3	3	8	75	61	28
-2	3	8	0	22	1
-1	3	8	135	127	8
0	3	8	63	70	17
1	3	8	0	24	1
2	3	8	0	7	1
3	3	8	155	151	7
4	3	8	296	290	6
5	3	8	109	118	8
6	3	8	0	29	1
7	3	8	30	38	30
8	3	8	45	66	44
9	3	8	153	134	21
10	3	8	79	101	44
11	3	8	102	80	37
-6	4	8	93	79	16
-5	4	8	0	2	1

h	k	l	10Fo	10Fc	10s
-4	4	8	72	66	20
-3	4	8	11	10	11
-2	4	8	0	4	1
-1	4	8	115	108	10
0	4	8	33	34	32
1	4	8	257	261	6
2	4	8	305	303	6
3	4	8	311	322	5
4	4	8	0	5	1
5	4	8	203	199	5
6	4	8	208	197	6
7	4	8	288	296	12
8	4	8	250	231	14
9	4	8	296	257	14
10	4	8	51	48	50
11	4	8	45	28	45
-5	5	8	0	5	1
-4	5	8	57	24	32
-3	5	8	78	77	18
-2	5	8	172	144	8
-1	5	8	0	35	1
0	5	8	0	19	1
1	5	8	33	44	33
2	5	8	364	357	12
3	5	8	81	69	12
4	5	8	130	139	18
5	5	8	41	22	40
6	5	8	122	89	39
7	5	8	133	142	16
8	5	8	170	162	18
9	5	8	133	116	28
10	5	8	224	220	17
11	5	8	26	10	26
-5	6	8	0	6	1
-4	6	8	32	2	32
-3	6	8	217	208	7
-2	6	8	52	51	29
-1	6	8	15	10	15
0	6	8	88	74	10
1	6	8	227	223	4
2	6	8	182	184	6
3	6	8	49	46	29
4	6	8	99	101	31
5	6	8	83	60	44
6	6	8	0	26	1
7	6	8	90	82	31
8	6	8	149	145	21
9	6	8	142	107	25
10	6	8	220	188	18
11	6	8	119	117	30
-4	7	8	0	54	1
-3	7	8	303	301	9
-2	7	8	194	184	11
-1	7	8	0	19	1
0	7	8	136	120	14
1	7	8	115	107	13
2	7	8	65	35	12
3	7	8	269	286	14
4	7	8	171	180	17
5	7	8	161	154	21
6	7	8	169	157	29
7	7	8	46	21	45
8	7	8	26	21	26
9	7	8	196	200	20
10	7	8	112	122	35
11	7	8	87	73	56
-4	8	8	23	32	23
-3	8	8	124	120	17
-2	8	8	146	138	15
-1	8	8	97	56	27
0	8	8	124	120	18
1	8	8	57	39	56
2	8	8	0	7	1
3	8	8	124	138	34
4	8	8	68	35	68
5	8	8	53	37	53
6	8	8	95	80	44
7	8	8	26	24	26
8	8	8	129	102	29
9	8	8	71	61	70
10	8	8	0	2	1
11	8	8	99	84	41
-3	9	8	34	15	34
-2	9	8	217	217	10
-1	9	8	104	70	20
0	9	8	81	78	80
1	9	8	0	17	1

h	k	l	10Fo	10Fc	10s
2	9	8	70	58	70
3	9	8	21	26	20
4	9	8	0	53	1
5	9	8	121	100	41
6	9	8	25	12	24
7	9	8	0	28	1
8	9	8	229	244	17
9	9	8	101	105	40
10	9	8	23	12	23
-2	10	8	116	86	18
-1	10	8	88	144	56
0	10	8	122	124	44
1	10	8	109	99	55
2	10	8	118	100	45
3	10	8	35	18	34
4	10	8	104	92	53
5	10	8	61	44	61
6	10	8	79	84	79
7	10	8	71	34	71
8	10	8	277	297	15
9	10	8	50	10	49
10	10	8	40	70	40
-1	11	8	28	54	27
0	11	8	0	8	1
1	11	8	81	73	81
2	11	8	23	67	23
3	11	8	93	92	88
4	11	8	38	8	37
5	11	8	34	15	33
6	11	8	173	210	26
7	11	8	68	56	67
8	11	8	156	148	27
9	11	8	115	126	33
0	12	8	112	104	48
1	12	8	60	39	60
2	12	8	0	34	1
3	12	8	56	30	56
4	12	8	164	168	31
5	12	8	93	91	69
6	12	8	71	24	71
7	12	8	23	34	23
8	12	8	0	19	1
2	13	8	97	86	68
3	13	8	50	32	50
4	13	8	129	95	80
5	13	8	70	74	70
6	13	8	26	8	25
7	13	8	0	73	1
0	-8	9	46	27	45
1	-8	9	9	30	9
-2	-7	9	29	53	29
-1	-7	9	118	116	21
0	-7	9	125	122	18
1	-7	9	43	53	42
2	-7	9	0	46	1
3	-7	9	137	123	18
4	-7	9	70	64	45
-3	-6	9	72	106	34
-2	-6	9	71	87	37
-1	-6	9	201	195	11
0	-6	9	177	174	14
1	-6	9	50	37	49
2	-6	9	51	60	50
3	-6	9	51	7	50
4	-6	9	38	22	38
5	-6	9	98	86	29
-4	-5	9	38	58	38
-3	-5	9	59	52	37
-2	-5	9	51	63	51
-1	-5	9	52	71	52
0	-5	9	84	90	35
1	-5	9	120	120	22
2	-5	9	56	67	56
3	-5	9	0	58	1
4	-5	9	99	89	27
5	-5	9	275	264	10
6	-5	9	57	54	56
7	-5	9	0	42	1
-5	-4	9	38	35	37
-4	-4	9	127	121	13
-3	-4	9	262	268	8
-2	-4	9	138	149	12
-1	-4	9	9	29	8
0	-4	9	129	126	22
1	-4	9	28	1	27
2	-4	9	0	17	1
3	-4	9	42	16	41

h	k	l	10Fo	10Fc	10s
4	-4	9	78	70	47
5	-4	9	11	4	11
6	-4	9	70	71	40
7	-4	9	73	44	35
-5	-3	9	29	4	28
-4	-3	9	97	102	17
-3	-3	9	101	111	18
-2	-3	9	77	74	24
-1	-3	9	123	113	13
0	-3	9	335	343	7
1	-3	9	128	122	17
2	-3	9	44	35	43
3	-3	9	134	122	19
4	-3	9	21	29	21
5	-3	9	170	165	13
6	-3	9	80	81	34
7	-3	9	0	22	1
8	-3	9	0	28	1
-5	-2	9	25	41	24
-4	-2	9	97	105	13
-3	-2	9	0	3	1
-2	-2	9	197	195	9
-1	-2	9	13	23	12
0	-2	9	38	37	38
1	-2	9	26	7	26
2	-2	9	103	93	22
3	-2	9	103	106	22
4	-2	9	15	8	15
5	-2	9	124	119	18
6	-2	9	80	67	31
7	-2	9	47	42	46
8	-2	9	56	61	56
9	-2	9	88	89	20
-6	-1	9	0	5	1
-5	-1	9	0	7	1
-4	-1	9	28	55	27
-3	-1	9	0	25	1
-2	-1	9	120	110	16
-1	-1	9	38	42	38
0	-1	9	107	95	17
1	-1	9	0	2	1
2	-1	9	0	12	1
3	-1	9	40	23	39
4	-1	9	16	23	16
5	-1	9	21	1	21
6	-1	9	85	82	23
7	-1	9	78	74	30
8	-1	9	24	4	24
9	-1	9	157	150	12
-6	0	9	146	132	20
-5	0	9	54	49	54
-4	0	9	74	77	43
-3	0	9	50	43	49
-2	0	9	110	105	14
-1	0	9	43	36	43
0	0	9	124	130	13
1	0	9	205	200	7
2	0	9	178	176	8
3	0	9	53	62	26
4	0	9	131	138	13
5	0	9	126	137	13
6	0	9	51	74	51
7	0	9	160	178	13
8	0	9	104	143	23
9	0	9	73	85	26
10	0	9	48	69	48
-6	1	9	131	109	19
-5	1	9	48	27	47
-4	1	9	64	68	64
-3	1	9	12	24	12
-2	1	9	115	102	15
-1	1	9	234	249	7
0	1	9	216	226	7
1	1	9	196	193	7
2	1	9	105	99	12
3	1	9	70	62	19
4	1	9	334	340	6
5	1	9	201	195	7
6	1	9	28	32	27
7	1	9	59	72	49
8	1	9	51	37	51
9	1	9	232	242	8
10	1	9	102	117	29
-5	2	9	102	103	26
-4	2	9	88	67	29
-3	2	9	65	67	64
-2	2	9	54	59	54

Table 6. . Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
-1	2	9	43	38	42
0	2	9	88	80	13
1	2	9	268	270	6
2	2	9	46	34	45
3	2	9	19	30	19
4	2	9	96	91	13
5	2	9	0	12	1
6	2	9	22	15	22
7	2	9	44	32	44
8	2	9	117	104	15
9	2	9	70	65	70
10	2	9	227	228	16
-5	3	9	46	6	45
-4	3	9	0	9	1
-3	3	9	68	54	30
-2	3	9	41	26	40
-1	3	9	156	148	8
0	3	9	339	330	9
1	3	9	7	22	6
2	3	9	70	77	19
3	3	9	119	114	10
4	3	9	125	128	9
5	3	9	219	211	6
6	3	9	54	61	34
7	3	9	73	66	33
8	3	9	227	236	16
9	3	9	0	35	1
10	3	9	87	92	39
11	3	9	38	43	37
-5	4	9	60	57	27
-4	4	9	101	110	13
-3	4	9	117	114	18
-2	4	9	193	188	10
-1	4	9	31	56	31
0	4	9	35	29	35
1	4	9	108	112	11
2	4	9	134	137	8
3	4	9	103	109	11
4	4	9	123	131	7
5	4	9	163	168	12
6	4	9	86	101	25
7	4	9	11	7	11
8	4	9	140	129	16
9	4	9	154	146	14
10	4	9	103	127	34
11	4	9	52	70	52
-5	5	9	92	77	17
-4	5	9	200	195	7
-3	5	9	104	96	14
-2	5	9	91	83	17
-1	5	9	37	64	37
0	5	9	24	16	23
1	5	9	0	23	1
2	5	9	0	1	1
3	5	9	197	201	6
4	5	9	21	11	21
5	5	9	121	119	17
6	5	9	92	93	40
7	5	9	29	5	29
8	5	9	57	55	56
9	5	9	215	216	16
10	5	9	96	117	39
-4	6	9	259	239	7
-3	6	9	124	124	11
-2	6	9	73	77	19
-1	6	9	48	28	21
0	6	9	0	11	1
1	6	9	306	311	6
2	6	9	188	175	7
3	6	9	87	80	23
4	6	9	94	92	47
5	6	9	164	177	17
6	6	9	57	89	56
7	6	9	170	153	28
8	6	9	133	103	27
9	6	9	118	81	30
10	6	9	162	148	22
11	6	9	111	96	30
-4	7	9	59	38	27
-3	7	9	51	53	47
-2	7	9	106	99	14
-1	7	9	212	180	7
0	7	9	70	53	18
1	7	9	106	95	8
2	7	9	211	207	17
3	7	9	81	72	63
4	7	9	202	212	18

h	k	l	10Fo	10Fc	10s
5	7	9	57	53	57
6	7	9	99	95	39
7	7	9	0	3	1
8	7	9	34	45	34
9	7	9	130	145	27
10	7	9	169	159	20
11	7	9	82	101	53
-3	8	9	100	94	19
-2	8	9	158	148	11
-1	8	9	48	43	48
0	8	9	199	211	22
1	8	9	37	15	36
2	8	9	183	174	24
3	8	9	262	286	16
4	8	9	180	176	24
5	8	9	154	134	29
6	8	9	82	50	67
7	8	9	10	23	10
8	8	9	92	75	45
9	8	9	76	90	75
10	8	9	100	79	40
-3	9	9	66	72	39
-2	9	9	15	12	15
-1	9	9	146	147	27
0	9	9	90	95	89
1	9	9	68	9	68
2	9	9	0	41	1
3	9	9	0	44	1
4	9	9	68	65	67
5	9	9	126	109	37
6	9	9	19	8	18
7	9	9	40	41	40
8	9	9	71	59	70
9	9	9	160	182	21
10	9	9	67	66	66
-2	10	9	66	52	66
-1	10	9	331	340	16
0	10	9	198	220	19
1	10	9	102	97	49
2	10	9	0	0	1
3	10	9	58	59	58
4	10	9	53	15	53
5	10	9	183	188	25
6	10	9	0	36	1
7	10	9	172	160	23
8	10	9	0	22	1
9	10	9	116	113	31
10	10	9	61	58	61
-1	11	9	69	58	69
0	11	9	210	235	22
1	11	9	198	199	21
2	11	9	41	49	41
3	11	9	139	88	36
4	11	9	181	213	31
5	11	9	112	112	47
6	11	9	69	32	68
7	11	9	112	76	43
8	11	9	121	91	61
9	11	9	95	133	52
1	12	9	179	157	26
2	12	9	95	77	57
3	12	9	114	112	43
4	12	9	40	31	39
5	12	9	82	63	81
6	12	9	41	52	41
7	12	9	0	11	1
8	12	9	47	49	46
3	13	9	30	17	29
4	13	9	130	104	44
5	13	9	132	139	36
6	13	9	42	62	42
1	-7	10	35	53	34
-2	-6	10	55	75	55
-1	-6	10	0	7	1
0	-6	10	64	48	45
1	-6	10	57	55	56
2	-6	10	148	129	17
3	-6	10	42	44	42
4	-6	10	132	115	19
-3	-5	10	20	18	19
-2	-5	10	119	138	16
-1	-5	10	122	125	17
0	-5	10	0	29	1
1	-5	10	99	105	23
2	-5	10	15	46	15
3	-5	10	17	31	17
4	-5	10	0	4	1

h	k	l	10Fo	10Fc	10s
5	-5	10	24	29	24
6	-5	10	45	46	45
-4	-4	10	18	25	17
-3	-4	10	82	78	21
-2	-4	10	90	87	18
-1	-4	10	35	58	34
0	-4	10	213	220	12
1	-4	10	45	57	45
2	-4	10	105	84	22
3	-4	10	81	81	35
4	-4	10	128	112	18
5	-4	10	115	97	20
6	-4	10	32	1	31
7	-4	10	0	24	1
-4	-3	10	24	14	24
-3	-3	10	48	56	48
-2	-3	10	121	125	14
-1	-3	10	250	258	8
0	-3	10	274	276	10
1	-3	10	61	53	60
2	-3	10	53	48	52
3	-3	10	0	30	1
4	-3	10	62	53	61
5	-3	10	117	129	21
6	-3	10	73	63	38
7	-3	10	162	148	14
8	-3	10	43	46	43
-4	-2	10	94	102	15
-3	-2	10	25	37	24
-2	-2	10	48	16	47
-1	-2	10	60	53	49
0	-2	10	131	123	13
1	-2	10	86	76	33
2	-2	10	52	63	51
3	-2	10	208	194	11
4	-2	10	132	119	18
5	-2	10	23	9	23
6	-2	10	115	132	20
7	-2	10	188	176	11
8	-2	10	0	29	1
-5	-1	10	59	39	58
-4	-1	10	0	9	1
-3	-1	10	98	86	16
-2	-1	10	240	246	8
-1	-1	10	169	168	11
0	-1	10	103	102	17
1	-1	10	3	0	3
2	-1	10	95	80	24
3	-1	10	54	63	54
4	-1	10	159	140	13
5	-1	10	160	153	13
6	-1	10	55	45	54
7	-1	10	58	51	46
8	-1	10	0	37	1
9	-1	10	155	163	12
-5	0	10	167	146	18
-4	0	10	49	38	49
-3	0	10	26	0	26
-2	0	10	108	88	17
-1	0	10	17	21	17
0	0	10	161	158	11
1	0	10	44	38	43
2	0	10	99	78	16
3	0	10	101	115	16
4	0	10	66	75	26
5	0	10	48	38	47
6	0	10	79	87	27
7	0	10	59	54	39
8	0	10	232	248	9
9	0	10	57	74	39
-5	1	10	31	29	31
-4	1	10	207	196	12
-3	1	10	48	18	48
-2	1	10	106	112	17
-1	1	10	119	119	13
0	1	10	19	50	19
1	1	10	16	35	15
2	1	10	51	62	30
3	1	10	159	151	10
4	1	10	156	141	10
5	1	10	67	53	30
6	1	10	136	121	13
7	1	10	0	1	1
8	1	10	115	109	16
9	1	10	0	18	1
10	1	10	38	18	37
-5	2	10	182	149	18

h	k	l	10Fo	10Fc	10s
-4	2	10	56	12	56
-3	2	10	0	38	1
-2	2	10	144	119	17
-1	2	10	0	4	1
0	2	10	50	58	31
1	2	10	100	88	12
2	2	10	194	189	7
3	2	10	171	171	8
4	2	10	241	240	6
5	2	10	124	120	9
6	2	10	106	101	15
7	2	10	68	67	67
8	2	10	44	12	43
9	2	10	48	3	47
10	2	10	128	167	29
-4	3	10	52	55	52
-3	3	10	0	26	1
-2	3	10	57	71	57
-1	3	10	61	52	27
0	3	10	41	53	41
1	3	10	28	23	28
2	3	10	39	43	38
3	3	10	180	174	8
4	3	10	135	138	10
5	3	10	67	93	22
6	3	10	162	163	13
7	3	10	190	192	18
8	3	10	51	29	51
9	3	10	108	91	31
10	3	10	73	62	56
-4	4	10	0	38	1
-3	4	10	199	172	11
-2	4	10	76	61	31
-1	4	10	65	35	65
0	4	10	80	70	19
1	4	10	131	139	9
2	4	10	17	38	17
3	4	10	45	41	45
4	4	10	136	137	16
5	4	10	58	43	57
6	4	10	146	142	16
7	4	10	156	155	14
8	4	10	187	179	12
9	4	10	254	230	17
10	4	10	73	55	59
-4	5	10	0	8	1
-3	5	10	163	163	12
-2	5	10	239	217	9
-1	5	10	68	111	22
0	5	10	42	31	41
1	5	10	0	36	1
2	5	10	163	173	10
3	5	10	47	57	46
4	5	10	42	19	42
5	5	10	29	11	29
6	5	10	0	0	1
7	5	10	77	72	40
8	5	10	111	114	23
9	5	10	84	69	29
10	5	10	183	180	20
-4	6	10	97	102	17
-3	6	10	126	95	13
-2	6	10	100	78	15
-1	6	10	51	63	51
0	6	10	57	50	36
1	6	10	63	58	22
2	6	10	53	37	53
3	6	10	122	117	17
4	6	10	32	15	31
5	6	10	64	64	64
6	6	10	0	11	1
7	6	10	0	4	1
8	6	10	179	173	19
9	6	10	72	82	71
10	6	10	74	76	51
-3	7	10	136	113	10
-2	7	10	0	13	1
-1	7	10	139	135	10
0	7	10	65	45	23
1	7	10	27	12	26
2	7	10	0	10	1
3	7	10	93	82	23
4	7	10	0	44	1
5	7	10	94	68	53
6	7	10	57	17	57
7	7	10	96	81	49
8	7	10	233	217	17

h	k	l	10Fo	10Fc	10s
9	7	10	46	13	46
10	7	10	85	72	38
-2	8	10	70	62	20
-1	8	10	41	9	40
0	8	10	0	25	1
1	8	10	50	30	49
2	8	10	18	9	17
3	8	10	0	7	1
4	8	10	200	197	20
5	8	10	195	188	24
6	8	10	79	75	79
7	8	10	151	135	29
8	8	10	87	71	56
9	8	10	94	96	39
10	8	10	52	31	51
-2	9	10	76	125	16
-1	9	10	0	4	1
0	9	10	59	87	59
1	9	10	36	15	36
2	9	10	149	138	30
3	9	10	140	131	31
4	9	10	136	146	35
5	9	10	98	93	78
6	9	10	34	4	34
7	9	10	178	187	23
8	9	10	138	147	28
9	9	10	126	126	26
10	9	10	0	31	1
-1	10	10	0	25	1
0	10	10	0	2	1
1	10	10	22	25	21
2	10	10	207	226	21
3	10	10	55	61	54
4	10	10	65	54	65
5	10	10	111	92	61
6	10	10	67	32	66
7	10	10	97	79	60
8	10	10	7	68	7
9	10	10	0	71	1
0	11	10	127	129	29
1	11	10	164	152	28
2	11	10	123	150	39
3	11	10	169	179	27
4	11	10	88	64	87
5	11	10	15	22	14
6	11	10	0	9	1
7	11	10	120	149	41
8	11	10	55	44	55
9	11	10	125	111	51
2	12	10	28	10	28
3	12	10	39	4	39
4	12	10	120	118	44
5	12	10	77	110	76
6	12	10	32	37	32
7	12	10	61	82	60
-1	-5	11	94	105	28
0	-5	11	31	22	30
1	-5	11	9	15	8
2	-5	11	50	48	50
3	-5	11	80	59	29
4	-5	11	123	104	18
-2	-4	11	88	99	23
-1	-4	11	0	2	1
0	-4	11	0	13	1
1	-4	11	42	23	42
2	-4	11	59	63	58
3	-4	11	0	8	1
4	-4	11	28	37	27
5	-4	11	79	76	29
6	-4	11	106	96	25
-3	-3	11	0	28	1
-2	-3	11	116	118	14
-1	-3	11	173	173	11
0	-3	11	71	64	31
1	-3	11	30	56	30
2	-3	11	65	55	47
3	-3	11	131	121	17
4	-3	11	41	51	40
5	-3	11	63	69	48
6	-3	11	83	98	30
7	-3	11	54	6	53
-3	-2	11	80	90	22
-2	-2	11	169	162	10
-1	-2	11	213	209	8
0	-2	11	244	252	9
1	-2	11	109	115	24
2	-2	11	66	71	51

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
3	-2	11	173	177	13
4	-2	11	61	44	61
5	-2	11	57	65	57
6	-2	11	31	29	30
7	-2	11	40	31	40
8	-2	11	10	40	10
-3	-1	11	73	69	24
-2	-1	11	37	34	37
-1	-1	11	188	180	10
0	-1	11	247	244	9
1	-1	11	224	222	10
2	-1	11	63	41	63
3	-1	11	46	54	46
4	-1	11	105	102	23
5	-1	11	35	2	35
6	-1	11	73	58	34
7	-1	11	182	187	11
8	-1	11	39	65	38
-4	0	11	111	138	23
-3	0	11	31	53	31
-2	0	11	79	59	22
-1	0	11	249	241	7
0	0	11	90	81	19
1	0	11	152	163	10
2	0	11	158	137	12
3	0	11	49	37	49
4	0	11	17	24	17
5	0	11	57	32	57
6	0	11	127	126	16
7	0	11	33	11	32
8	0	11	71	61	28
9	0	11	64	4	63
-4	1	11	101	62	30
-3	1	11	72	18	55
-2	1	11	173	149	16
-1	1	11	174	193	10
0	1	11	0	2	1
1	1	11	35	15	34
2	1	11	30	43	29
3	1	11	170	165	9
4	1	11	183	172	10
5	1	11	63	51	49
6	1	11	154	146	12
7	1	11	40	4	40
8	1	11	0	13	1
9	1	11	0	31	1
-4	2	11	78	41	40
-3	2	11	35	40	35
-2	2	11	45	56	44
-1	2	11	30	24	30
0	2	11	0	43	1
1	2	11	122	128	12
2	2	11	141	129	11
3	2	11	98	88	14
4	2	11	140	130	11
5	2	11	37	52	37
6	2	11	64	63	64
7	2	11	159	152	21
8	2	11	19	19	18
9	2	11	44	53	44
10	2	11	128	120	26
-4	3	11	157	131	20
-3	3	11	181	185	15
-2	3	11	0	0	1
-1	3	11	63	64	41
0	3	11	65	71	24
1	3	11	110	98	14
2	3	11	35	32	35
3	3	11	103	87	16
4	3	11	226	229	7
5	3	11	108	124	21
6	3	11	85	73	42
7	3	11	210	211	17
8	3	11	74	66	74
9	3	11	147	120	39
10	3	11	35	14	35
-3	4	11	7	51	7
-2	4	11	73	65	29
-1	4	11	70	69	35
0	4	11	94	103	17
1	4	11	45	48	44
2	4	11	156	153	9
3	4	11	184	161	14
4	4	11	148	159	13
5	4	11	49	64	49
6	4	11	210	193	12
7	4	11	0	15	1
8	4	11	69	67	68
9	4	11	248	247	17
10	4	11	0	17	1
-3	5	11	0	33	1
-2	5	11	181	177	11
-1	5	11	60	86	60
0	5	11	75	74	25
1	5	11	0	10	1
2	5	11	38	41	38
3	5	11	77	95	30
4	5	11	0	24	1
5	5	11	26	35	26
6	5	11	207	211	12
7	5	11	0	1	1
8	5	11	49	26	49
9	5	11	103	122	45
10	5	11	22	1	22
-3	6	11	48	30	47
-2	6	11	0	13	1
-1	6	11	119	130	15
0	6	11	109	103	18
1	6	11	182	194	10
2	6	11	146	155	13
3	6	11	78	68	29
4	6	11	197	194	12
5	6	11	115	110	20
6	6	11	156	164	17
7	6	11	133	127	20
8	6	11	134	120	22
9	6	11	66	66	66
10	6	11	0	23	1
-2	7	11	22	9	21
-1	7	11	69	80	32
0	7	11	154	157	22
1	7	11	171	153	25
2	7	11	155	158	14
3	7	11	69	79	35
4	7	11	78	73	32
5	7	11	129	124	20
6	7	11	0	37	1
7	7	11	61	41	60
8	7	11	82	64	82
9	7	11	128	117	29
10	7	11	23	22	23
-2	8	11	7	10	6
-1	8	11	29	36	28
0	8	11	96	117	39
1	8	11	41	44	40
2	8	11	149	151	14
3	8	11	215	215	10
4	8	11	150	148	16
5	8	11	105	116	43
6	8	11	107	98	55
7	8	11	77	82	76
8	8	11	48	57	48
9	8	11	53	63	53
10	8	11	129	100	39
-1	9	11	17	17	16
0	9	11	159	180	23
1	9	11	118	86	52
2	9	11	127	129	29
3	9	11	132	127	17
4	9	11	77	74	76
5	9	11	78	38	78
6	9	11	0	25	1
7	9	11	0	1	1
8	9	11	0	26	1
9	9	11	159	163	27
0	10	11	58	33	58
1	10	11	0	2	1
2	10	11	131	115	28
3	10	11	73	64	72
4	10	11	192	185	21
5	10	11	67	45	66
6	10	11	49	40	48
7	10	11	48	0	47
8	10	11	0	2	1
9	10	11	0	5	1
1	11	11	169	165	27
2	11	11	60	35	59
3	11	11	72	78	72
4	11	11	179	178	24
5	11	11	0	9	1
6	11	11	222	232	22
7	11	11	103	91	53
8	11	11	42	36	41
3	12	11	21	53	21
4	12	11	78	65	78
5	12	11	91	88	65
6	12	11	59	44	59
0	-4	12	39	27	38
1	-4	12	31	29	31
2	-4	12	210	228	11
3	-4	12	140	124	17
4	-4	12	0	23	1
-1	-3	12	33	2	32
0	-3	12	100	88	20
1	-3	12	118	120	17
2	-3	12	55	67	55
3	-3	12	192	189	11
4	-3	12	111	86	19
5	-3	12	14	4	14
-2	-2	12	28	30	27
-1	-2	12	0	35	1
0	-2	12	19	59	19
1	-2	12	87	90	28
2	-2	12	44	47	43
3	-2	12	40	32	40
4	-2	12	72	76	31
5	-2	12	167	147	15
6	-2	12	204	195	11
7	-2	12	105	116	21
-2	-1	12	178	183	9
-1	-1	12	29	35	28
0	-1	12	0	14	1
1	-1	12	67	83	35
2	-1	12	114	120	20
3	-1	12	0	17	1
4	-1	12	25	9	25
5	-1	12	141	134	14
6	-1	12	34	28	33
7	-1	12	39	60	38
-2	0	12	145	125	11
-1	0	12	53	3	52
0	0	12	143	128	11
1	0	12	0	15	1
2	0	12	203	202	11
3	0	12	0	22	1
4	0	12	45	68	45
5	0	12	0	17	1
6	0	12	70	62	32
7	0	12	125	148	28
8	0	12	89	75	46
-3	1	12	51	10	50
-2	1	12	240	221	9
-1	1	12	25	42	24
0	1	12	177	178	10
1	1	12	196	206	8
2	1	12	256	272	15
3	1	12	36	33	36
4	1	12	99	84	23
5	1	12	133	108	15
6	1	12	55	64	54
7	1	12	18	30	17
8	1	12	38	38	38
9	1	12	28	20	28
-3	2	12	90	54	26
-2	2	12	88	102	33
-1	2	12	0	23	1
0	2	12	117	115	15
1	2	12	0	12	1
2	2	12	217	220	7
3	2	12	98	90	17
4	2	12	84	60	23
5	2	12	59	58	59
6	2	12	129	143	25
7	2	12	117	104	28
8	2	12	165	127	26
9	2	12	71	75	71
-2	3	12	31	67	31
-1	3	12	59	76	58
0	3	12	10	50	10
1	3	12	136	155	10
2	3	12	135	128	11
3	3	12	112	121	18
4	3	12	110	131	21
5	3	12	0	8	1
6	3	12	116	128	28
7	3	12	29	33	29
8	3	12	178	178	19
9	3	12	29	26	29
-2	4	12	59	37	58
-1	4	12	57	45	57
0	4	12	95	85	20
1	4	12	188	179	8
2	4	12	208	208	9
3	4	12	205	213	11
4	4	12	53	52	53
5	4	12	9	47	8
6	4	12	85	78	28
7	4	12	116	115	31
8	4	12	127	102	28
9	4	12	45	57	45
-2	5	12	29	4	28
-1	5	12	0	10	1
0	5	12	52	25	51
1	5	12	133	119	16
2	5	12	43	82	43
3	5	12	0	2	1
4	5	12	12	3	12
5	5	12	76	89	34
6	5	12	168	171	14
7	5	12	42	10	41
8	5	12	122	112	32
9	5	12	25	14	24
-2	6	12	74	77	32
-1	6	12	0	15	1
0	6	12	37	20	36
1	6	12	18	40	18
2	6	12	6	7	6
3	6	12	196	203	11
4	6	12	39	57	38
5	6	12	0	3	1
6	6	12	62	50	62
7	6	12	142	145	19
8	6	12	127	132	21
9	6	12	133	82	35
-1	7	12	118	119	30
0	7	12	55	57	55
1	7	12	114	119	31
2	7	12	189	199	10
3	7	12	146	173	25
4	7	12	9	15	8
5	7	12	0	27	1
6	7	12	58	59	58
7	7	12	165	142	18
8	7	12	227	224	12
9	7	12	89	82	33
-1	8	12	0	25	1
0	8	12	34	10	34
1	8	12	169	148	22
2	8	12	49	22	48
3	8	12	27	2	27
4	8	12	147	144	15
5	8	12	51	60	51
6	8	12	0	21	1
7	8	12	52	91	52
8	8	12	0	29	1
9	8	12	72	47	72
0	9	12	56	39	55
1	9	12	187	154	20
2	9	12	80	82	60
3	9	12	146	165	13
4	9	12	41	12	41
5	9	12	195	181	18
6	9	12	155	167	23
7	9	12	83	46	83
8	9	12	28	21	27
9	9	12	61	70	60
1	10	12	104	89	40
2	10	12	24	7	24
3	10	12	39	15	38
4	10	12	0	55	1
5	10	12	112	111	33
6	10	12	23	51	22
7	10	12	0	29	1
8	10	12	55	31	55
3	11	12	176	185	21
4	11	12	0	13	1
5	11	12	0	33	1
6	11	12	34	65	33
7	11	12	101	85	66
1	-2	13	88	108	22
2	-2	13	61	69	42
3	-2	13	96	122	20
4	-2	13	57	5	56
5	-2	13	184	172	11
0	-1	13	149	161	13
1	-1	13	30	44	30
2	-1	13	190	186	10
3	-1	13	168	162	12
4	-1	13	88	81	2
5	-1	13	9	22	[illegible]
6	-1	13	20	14	1
-1	0	13	64	66	31
0	0	13	61	39	28
1	0	13	30	48	29
2	0	13	0	31	1
3	0	13	69	72	31
4	0	13	95	98	38
5	0	13	23	38	22
6	0	13	0	42	1
7	0	13	19	42	19
-1	1	13	107	109	19
0	1	13	180	185	9
1	1	13	0	17	1
2	1	13	128	187	22
3	1	13	144	124	15
4	1	13	60	46	51
5	1	13	39	5	38
6	1	13	0	42	1
7	1	13	54	43	53
8	1	13	48	69	48
-1	2	13	23	47	23
0	2	13	0	34	1
1	2	13	41	55	40
2	2	13	175	185	9
3	2	13	286	286	15
4	2	13	93	61	46
5	2	13	116	115	29
6	2	13	104	110	30
7	2	13	80	89	51
8	2	13	33	48	32
-1	3	13	69	64	29
0	3	13	0	49	1
1	3	13	47	59	46
2	3	13	104	104	19
3	3	13	60	55	59
4	3	13	60	49	60
5	3	13	52	74	52
6	3	13	228	208	17
7	3	13	60	87	59
8	3	13	0	39	1
-1	4	13	60	59	41
0	4	13	0	1	1
1	4	13	89	98	20
2	4	13	190	188	11
3	4	13	39	14	38
4	4	13	194	191	12
5	4	13	91	81	25
6	4	13	67	77	67
7	4	13	52	48	52
8	4	13	42	46	42
9	4	13	0	33	1
-1	5	13	28	87	27
0	5	13	159	146	16
1	5	13	0	19	1
2	5	13	39	69	39
3	5	13	52	63	52
4	5	13	57	68	57
5	5	13	161	161	13
6	5	13	101	112	21
7	5	13	75	77	51
8	5	13	58	60	57
9	5	13	49	56	48
0	6	13	184	181	18
1	6	13	140	139	24
2	6	13	47	48	46
3	6	13	39	38	39
4	6	13	16	4	15
5	6	13	202	211	11
6	6	13	27	33	26
7	6	13	55	47	54
8	6	13	121	118	29
9	6	13	92	114	51
0	7	13	96	101	32
1	7	13	106	110	24
2	7	13	152	157	22
3	7	13	0	14	1
4	7	13	0	13	1
5	7	13	24	2	23
6	7	13	71	70	42
7	7	13	121	117	22
8	7	13	130	125	20
9	7	13	0	33	1
1	8	13	78	69	38
2	8	13	99	89	36
3	8	13	43	45	42

Table 6. Observed and calculated structure factors for af011

h	k	l	10Fo	10Fc	10s
4	8	13	105	114	20
5	8	13	35	37	34
6	8	13	35	46	34
7	8	13	20	41	19
8	8	13	76	73	51
2	9	13	96	114	39
3	9	13	149	166	25
4	9	13	30	12	29
5	9	13	75	71	38
6	9	13	83	82	32
7	9	13	44	12	43
8	9	13	0	31	1
3	10	13	70	71	70
4	10	13	144	114	26
5	10	13	51	30	51
6	10	13	57	3	57
7	10	13	0	23	1
2	0	14	62	81	42
3	0	14	0	30	1
4	0	14	72	82	71
5	0	14	53	64	52
1	1	14	153	159	11
2	1	14	57	83	57
3	1	14	0	46	1
4	1	14	46	38	46
5	1	14	0	33	1
6	1	14	64	71	64
1	2	14	45	46	44
2	2	14	96	103	39
3	2	14	124	132	26
4	2	14	79	55	49
5	2	14	142	156	25
6	2	14	142	134	23
7	2	14	61	39	60
1	3	14	41	41	40
2	3	14	26	34	26
3	3	14	80	95	43
4	3	14	37	50	36
5	3	14	98	101	31
6	3	14	87	88	39
7	3	14	63	64	63
1	4	14	0	3	1
2	4	14	48	25	48
3	4	14	144	159	13
4	4	14	73	63	42
5	4	14	128	144	25
6	4	14	0	43	1
7	4	14	0	13	1
1	5	14	37	6	36
2	5	14	0	18	1
3	5	14	126	127	14
4	5	14	32	29	32
5	5	14	122	127	18
6	5	14	0	20	1
7	5	14	0	55	1
1	6	14	58	44	58
2	6	14	64	84	64
3	6	14	0	21	1
4	6	14	103	112	19
5	6	14	0	9	1
6	6	14	97	102	22
7	6	14	46	39	46
2	7	14	90	52	32
3	7	14	59	87	35
4	7	14	70	70	32
5	7	14	48	65	48
6	7	14	23	53	22
7	7	14	65	67	64
3	8	14	57	76	57
4	8	14	28	26	27
5	8	14	74	80	36
6	8	14	22	33	21
7	8	14	21	6	20
5	9	14	53	57	53
4	4	15	146	162	22
4	5	15	65	55	31
5	5	15	67	88	66

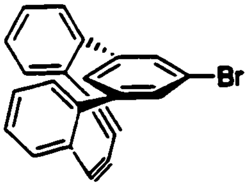
Br

Table 1. Crystal data and structure refinement for af012.

Identification code	af012	
Empirical formula	C22 H11 Br	
Formula weight	355.22	
Temperature	203(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 3.9174(4) A	alpha = 91.613(2) deg.
	b = 10.510(1) A	beta = 92.290(2) deg.
	c = 18.179(2) A	gamma = 93.982(2) deg.
Volume	745.7(1) A^3	
Z, Calculated density	2, 1.582 Mg/m^3	
Absorption coefficient	2.752 mm^-1	
F(000)	356	
Crystal size	0.40 x 0.10 x 0.10 mm	
Theta range for data collection	1.94 to 28.93 deg.	
Limiting indices	-5<=h<=5, -13<=k<=13, 0<=l<=24	
Reflections collected / unique	5844 / 3433 [R(int) = 0.0464]	
Completeness to theta = 28.93	86.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.928078 and 0.495610	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3433 / 0 / 208	
Goodness-of-fit on F^2	1.014	
Final R indices [I>2sigma(I)]	R1 = 0.0447, wR2 = 0.0983	
R indices (all data)	R1 = 0.0531, wR2 = 0.0993	
Largest diff. peak and hole	0.836 and -1.097 e.A^-3	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for af012.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Br	-589(1)	2152(1)	620(1)	28(1)
C(1)	4481(7)	5(3)	2532(2)	22(1)
C(2)	3818(7)	1291(3)	2645(2)	22(1)
C(3)	2280(7)	1917(3)	2068(2)	24(1)
C(4)	1443(7)	1273(3)	1408(2)	21(1)
C(5)	1958(7)	-12(3)	1307(2)	23(1)
C(6)	3467(7)	-664(3)	1877(2)	21(1)
C(7)	3899(7)	-2059(3)	1757(2)	22(1)
C(8)	5164(8)	-2495(3)	1096(2)	27(1)
C(9)	5516(8)	-3771(3)	957(2)	32(1)
C(10)	4560(9)	-4669(3)	1465(2)	35(1)
C(11)	3248(9)	-4274(3)	2117(2)	33(1)
C(12)	2912(8)	-2980(3)	2281(2)	26(1)
C(13)	1638(8)	-2548(3)	2966(2)	28(1)
C(14)	970(8)	-1914(3)	3493(2)	29(1)
C(15)	1164(8)	-807(3)	3922(2)	30(1)
C(16)	2149(8)	288(3)	4063(2)	28(1)
C(17)	3750(8)	1547(3)	4048(2)	27(1)
C(18)	4532(9)	2291(3)	4686(2)	33(1)
C(19)	6205(9)	3494(4)	4658(2)	37(1)
C(20)	7078(9)	3964(3)	3981(2)	34(1)
C(21)	6319(8)	3237(3)	3343(2)	29(1)
C(22)	4687(8)	2022(3)	3357(2)	24(1)

Table 3. Bond lengths [A] and angles [deg] for af012.

Br-C(4)	1.903(3)
C(1)-C(6)	1.395(5)
C(1)-C(2)	1.405(4)
C(2)-C(3)	1.393(4)
C(2)-C(22)	1.501(5)
C(3)-C(4)	1.377(5)
C(4)-C(5)	1.388(4)
C(5)-C(6)	1.394(4)
C(6)-C(7)	1.498(4)
C(7)-C(8)	1.395(5)
C(7)-C(12)	1.422(5)
C(8)-C(9)	1.376(5)
C(9)-C(10)	1.383(5)
C(10)-C(11)	1.375(5)
C(11)-C(12)	1.400(5)
C(12)-C(13)	1.434(5)
C(13)-C(14)	1.200(5)
C(14)-C(15)	1.377(5)
C(15)-C(16)	1.206(5)
C(16)-C(17)	1.425(5)
C(17)-C(18)	1.393(5)
C(17)-C(22)	1.417(5)
C(18)-C(19)	1.386(5)
C(19)-C(20)	1.386(6)
C(20)-C(21)	1.380(5)
C(21)-C(22)	1.389(5)

C(6)-C(1)-C(2)	121.6(3)
C(3)-C(2)-C(1)	118.3(3)
C(3)-C(2)-C(22)	118.5(3)
C(1)-C(2)-C(22)	123.2(3)
C(4)-C(3)-C(2)	120.0(3)
C(3)-C(4)-C(5)	121.8(3)
C(3)-C(4)-Br	119.3(2)
C(5)-C(4)-Br	118.9(2)
C(4)-C(5)-C(6)	119.4(3)
C(5)-C(6)-C(1)	118.8(3)
C(5)-C(6)-C(7)	118.1(3)
C(1)-C(6)-C(7)	123.2(3)
C(8)-C(7)-C(12)	117.9(3)
C(8)-C(7)-C(6)	119.6(3)
C(12)-C(7)-C(6)	122.4(3)
C(9)-C(8)-C(7)	121.4(3)
C(8)-C(9)-C(10)	120.7(4)
C(11)-C(10)-C(9)	119.3(3)
C(10)-C(11)-C(12)	121.3(3)
C(11)-C(12)-C(7)	119.2(3)
C(11)-C(12)-C(13)	122.1(3)
C(7)-C(12)-C(13)	118.6(3)
C(14)-C(13)-C(12)	164.1(3)
C(13)-C(14)-C(15)	153.5(4)
C(16)-C(15)-C(14)	153.4(4)
C(15)-C(16)-C(17)	164.6(4)
C(18)-C(17)-C(22)	119.5(3)
C(18)-C(17)-C(16)	122.3(3)
C(22)-C(17)-C(16)	118.1(3)

C(19)-C(18)-C(17)	121.1(4)
C(20)-C(19)-C(18)	119.2(4)
C(21)-C(20)-C(19)	120.3(4)
C(20)-C(21)-C(22)	121.6(4)
C(21)-C(22)-C(17)	118.2(3)
C(21)-C(22)-C(2)	119.4(3)
C(17)-C(22)-C(2)	122.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for af012.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Br	29(1)	33(1)	23(1)	9(1)	-1(1)	5(1)
C(1)	19(1)	23(2)	24(2)	5(1)	3(1)	1(1)
C(2)	20(1)	26(2)	22(2)	2(1)	6(1)	1(1)
C(3)	23(2)	21(2)	27(2)	5(1)	7(1)	2(1)
C(4)	19(1)	27(2)	19(2)	9(1)	4(1)	1(1)
C(5)	24(2)	27(2)	18(2)	1(1)	1(1)	0(1)
C(6)	20(1)	22(2)	22(2)	5(1)	6(1)	-2(1)
C(7)	18(1)	24(2)	25(2)	3(1)	-1(1)	1(1)
C(8)	24(2)	27(2)	30(2)	4(2)	-1(1)	-5(1)
C(9)	29(2)	29(2)	37(2)	-6(2)	4(2)	-1(1)
C(10)	35(2)	23(2)	47(3)	-1(2)	2(2)	2(1)
C(11)	32(2)	27(2)	40(2)	7(2)	2(2)	-1(1)
C(12)	23(2)	27(2)	27(2)	5(1)	-3(1)	0(1)
C(13)	27(2)	26(2)	31(2)	12(2)	3(2)	-1(1)
C(14)	29(2)	31(2)	27(2)	10(2)	3(2)	1(1)
C(15)	29(2)	40(2)	22(2)	10(2)	5(1)	6(1)
C(16)	29(2)	41(2)	16(2)	4(2)	8(1)	9(1)
C(17)	25(2)	32(2)	23(2)	1(1)	-1(1)	8(1)
C(18)	37(2)	42(2)	21(2)	-2(2)	-2(2)	13(2)
C(19)	39(2)	42(2)	30(2)	-13(2)	-8(2)	10(2)
C(20)	31(2)	31(2)	39(2)	-5(2)	-4(2)	3(1)
C(21)	28(2)	31(2)	30(2)	-1(2)	2(2)	5(1)
C(22)	22(2)	29(2)	23(2)	2(1)	2(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for af012.

	x	y	z	U(eq)
H(1A)	5636	-414	2907	27
H(3A)	1813	2778	2129	28
H(5A)	1294	-440	857	27
H(8A)	5788	-1903	738	33
H(9A)	6417	-4036	510	38
H(10A)	4805	-5539	1366	42
H(11A)	2562	-4885	2459	40
H(18A)	3914	1973	5144	39
H(19A)	6741	3984	5092	44
H(20A)	8194	4782	3955	41
H(21A)	6920	3573	2888	35

Table 6. Observed and calculated structure factors for af012

h	k	l	10Fo	10Fc	10s
1	0	0	196	185	4
2	0	0	304	301	7
3	0	0	71	66	12
4	0	0	55	55	16
5	0	0	25	21	25
-5	1	0	41	28	40
-4	1	0	331	341	13
-3	1	0	322	319	8
-2	1	0	251	250	5
-1	1	0	104	94	4
0	1	0	448	485	13
1	1	0	692	684	15
2	1	0	345	330	8
3	1	0	176	180	6
4	1	0	237	238	7
5	1	0	102	87	20
-5	2	0	18	26	18
-4	2	0	99	95	8
-3	2	0	258	260	7
-2	2	0	705	696	13
-1	2	0	56	55	6
0	2	0	724	772	16
1	2	0	603	630	14
2	2	0	56	52	9
3	2	0	27	30	27
4	2	0	77	76	11
-5	3	0	86	89	11
-4	3	0	142	145	7
-3	3	0	168	164	5
-2	3	0	11	11	11
-1	3	0	219	210	3
0	3	0	299	372	29
1	3	0	426	386	8
2	3	0	553	549	10
3	3	0	230	245	6
4	3	0	77	71	19
-5	4	0	116	81	24
-4	4	0	261	266	7
-3	4	0	87	94	6
-2	4	0	470	522	20
-1	4	0	247	251	6
0	4	0	409	427	11
1	4	0	252	235	5
2	4	0	46	38	16
3	4	0	220	230	6
4	4	0	34	27	33
-4	5	0	7	10	6
-3	5	0	121	114	5
-2	5	0	170	154	4
-1	5	0	34	28	33
0	5	0	644	669	11
1	5	0	398	404	7
2	5	0	311	317	7
3	5	0	158	157	6
4	5	0	127	125	7
-4	6	0	206	229	9
-3	6	0	275	292	7
-2	6	0	292	292	7
-1	6	0	274	269	7
0	6	0	40	44	11
1	6	0	21	18	20
2	6	0	130	130	6
3	6	0	195	202	6
4	6	0	157	152	7
-4	7	0	0	2	1
-3	7	0	173	188	6
-2	7	0	214	213	4
-1	7	0	263	256	7
0	7	0	356	352	8
1	7	0	251	249	6
2	7	0	340	335	8
3	7	0	10	23	10
4	7	0	8	10	7
-4	8	0	111	120	7
-3	8	0	115	128	6
-2	8	0	145	150	4
-1	8	0	85	79	8
0	8	0	47	31	19
1	8	0	211	205	9
2	8	0	88	84	8
3	8	0	303	308	8
4	8	0	69	55	27
-4	9	0	87	84	12
-3	9	0	132	135	7
-2	9	0	205	211	4
-1	9	0	287	293	7
0	9	0	300	290	7
1	9	0	42	25	42
2	9	0	158	160	9
3	9	0	67	74	20
-3	10	0	67	57	14
-2	10	0	0	7	1
-1	10	0	108	107	7
0	10	0	174	175	9
1	10	0	137	130	10
2	10	0	72	72	16
3	10	0	78	77	20
-3	11	0	174	175	7
-2	11	0	113	117	4
-1	11	0	93	92	8
0	11	0	97	93	12
1	11	0	82	81	14
2	11	0	0	5	1
-2	12	0	52	56	9
-1	12	0	59	48	27
0	12	0	128	129	10
1	12	0	129	129	10
2	12	0	95	91	15
-2	13	0	105	103	12
-1	13	0	28	16	28
0	13	0	61	53	26
1	13	0	26	36	26
-1	-13	1	109	97	13
0	-13	1	29	2	29
1	-13	1	36	43	36
2	-13	1	49	42	49
-2	-12	1	54	56	41
-1	-12	1	162	148	10
0	-12	1	170	167	9
1	-12	1	126	115	11
2	-12	1	138	147	4
-2	-11	1	42	42	41
-1	-11	1	0	4	1
0	-11	1	0	24	1
1	-11	1	187	178	6
2	-11	1	189	191	4
3	-11	1	161	168	7
-3	-10	1	92	93	13
-2	-10	1	137	143	9
-1	-10	1	228	214	10
0	-10	1	275	279	10
1	-10	1	57	47	15
2	-10	1	30	37	29
3	-10	1	27	29	26
-3	-9	1	37	45	36
-2	-9	1	52	55	30
-1	-9	1	199	190	9
0	-9	1	239	226	7
1	-9	1	266	261	7
2	-9	1	110	115	4
3	-9	1	216	226	7
4	-9	1	59	57	30
-4	-8	1	150	144	13
-3	-8	1	191	190	7
-2	-8	1	86	85	10
-1	-8	1	226	210	9
0	-8	1	95	82	7
1	-8	1	52	45	18
2	-8	1	44	40	10
3	-8	1	16	23	16
4	-8	1	119	124	7
-4	-7	1	33	10	32
-3	-7	1	101	107	7
-2	-7	1	153	149	6
-1	-7	1	77	74	8
0	-7	1	197	230	12
1	-7	1	475	456	10
2	-7	1	225	229	4
3	-7	1	224	236	6
4	-7	1	19	15	18
-4	-6	1	185	173	8
-3	-6	1	109	108	7
-2	-6	1	162	166	6
-1	-6	1	245	242	5
0	-6	1	18	8	17
1	-6	1	17	15	16
2	-6	1	329	312	7
3	-6	1	256	265	7
4	-6	1	40	51	28
-4	-5	1	0	4	1
-3	-5	1	133	133	6
-2	-5	1	203	209	6
-1	-5	1	637	635	11
0	-5	1	54	71	7
1	-5	1	391	381	9
2	-5	1	334	316	7
3	-5	1	23	26	22
4	-5	1	119	126	6
-4	-4	1	208	217	7
-3	-4	1	193	197	6
-2	-4	1	42	41	12
-1	-4	1	38	31	12
0	-4	1	177	183	4
1	-4	1	105	110	4
2	-4	1	264	274	5
3	-4	1	449	463	8
4	-4	1	157	155	7
5	-4	1	78	66	26
-4	-3	1	95	94	8
-3	-3	1	381	394	9
-2	-3	1	227	237	6
-1	-3	1	644	606	12
0	-3	1	467	479	8
1	-3	1	37	35	8
2	-3	1	252	242	5
3	-3	1	120	120	5
4	-3	1	68	59	14
5	-3	1	94	79	20
-4	-2	1	94	96	8
-3	-2	1	105	106	5
-2	-2	1	270	264	5
-1	-2	1	273	274	5
0	-2	1	77	70	5
1	-2	1	856	885	15
2	-2	1	67	65	7
3	-2	1	392	386	7
4	-2	1	121	126	7
5	-2	1	51	52	27
-5	-1	1	110	111	15
-4	-1	1	107	103	8
-3	-1	1	407	397	8
-2	-1	1	186	187	5
-1	-1	1	706	703	13
0	-1	1	236	238	5
1	-1	1	106	96	4
2	-1	1	76	80	5
3	-1	1	164	162	6
4	-1	1	95	98	8
5	-1	1	72	71	15
-5	0	1	94	88	13
-4	0	1	75	73	12
-3	0	1	46	38	21
-2	0	1	232	228	6
-1	0	1	1073	1119	23
1	0	1	161	140	4
2	0	1	459	447	8
3	0	1	105	103	7
4	0	1	172	182	6
5	0	1	32	31	32
-5	1	1	149	156	7
-4	1	1	244	261	7
-3	1	1	186	184	6
-2	1	1	343	342	8
-1	1	1	361	338	6
0	1	1	101	124	6
1	1	1	230	217	5
2	1	1	70	83	8
3	1	1	199	200	6
4	1	1	196	199	7
-5	2	1	34	16	34
-4	2	1	82	86	9
-3	2	1	112	114	7
-2	2	1	81	79	5
-1	2	1	717	713	13
0	2	1	560	627	10
1	2	1	292	303	5
2	2	1	397	377	7
3	2	1	70	65	11
4	2	1	58	55	15
-5	3	1	122	118	14
-4	3	1	199	205	6
-3	3	1	153	156	6
-2	3	1	386	365	7
-1	3	1	112	99	4
0	3	1	234	226	4
1	3	1	262	263	5
2	3	1	308	303	6
3	3	1	183	189	6
4	3	1	134	141	7
-5	4	1	58	42	47
-4	4	1	56	49	18
-3	4	1	326	333	6
-2	4	1	177	180	4
-1	4	1	576	560	12
0	4	1	569	581	10
1	4	1	91	100	4
2	4	1	133	133	5
3	4	1	37	33	36
4	4	1	46	49	23
-4	5	1	242	248	7
-3	5	1	68	63	10
-2	5	1	11	1	10
-1	5	1	31	16	31
0	5	1	611	587	11
1	5	1	293	300	5
2	5	1	232	236	6
3	5	1	220	221	10
4	5	1	84	74	10
-4	6	1	227	244	9
-3	6	1	249	255	7
-2	6	1	318	322	4
-1	6	1	204	208	6
0	6	1	252	254	5
1	6	1	22	14	21
2	6	1	75	71	11
3	6	1	78	70	11
4	6	1	52	39	20
-4	7	1	33	33	33
-3	7	1	18	13	18
-2	7	1	118	115	4
-1	7	1	192	193	6
0	7	1	257	264	5
1	7	1	258	254	7
2	7	1	206	198	6
3	7	1	114	110	7
4	7	1	86	83	12
-4	8	1	81	95	12
-3	8	1	140	156	6
-2	8	1	239	255	4
-1	8	1	101	103	7
0	8	1	86	75	7
1	8	1	48	31	47
2	8	1	106	98	7
3	8	1	180	186	7
4	8	1	35	15	34
-4	9	1	11	25	11
-3	9	1	35	38	35
-2	9	1	107	115	4
-1	9	1	236	246	6
0	9	1	226	211	9
1	9	1	214	213	9
2	9	1	168	159	9
3	9	1	67	60	22
-3	10	1	76	70	12
-2	10	1	149	160	4
-1	10	1	18	6	17
0	10	1	42	42	42
1	10	1	114	116	11
2	10	1	72	75	12
3	10	1	120	120	11
-3	11	1	80	80	11
-2	11	1	104	109	4
-1	11	1	203	195	7
0	11	1	169	165	9
1	11	1	190	179	10
2	11	1	56	40	36
-2	12	1	23	22	22
-1	12	1	52	33	38
0	12	1	119	116	11
1	12	1	81	77	17
-2	13	1	113	99	11
-1	13	1	116	103	13
0	13	1	90	79	17
1	13	1	56	38	37
-1	-13	2	80	64	19
0	-13	2	34	39	33
1	-13	2	11	16	11
2	-13	2	38	31	37
-2	-12	2	110	98	13
-1	-12	2	133	119	10
0	-12	2	86	73	15
1	-12	2	202	191	7
2	-12	2	77	79	6
-2	-11	2	57	62	25
-1	-11	2	83	87	14
0	-11	2	36	28	36
1	-11	2	65	56	14
2	-11	2	104	105	5
3	-11	2	17	20	17
-3	-10	2	42	47	42
-2	-10	2	113	118	9
-1	-10	2	249	253	10
0	-10	2	129	125	10
1	-10	2	166	160	6
2	-10	2	147	152	4
3	-10	2	59	55	16
-3	-9	2	33	21	32
-2	-9	2	187	190	9
-1	-9	2	120	117	10
0	-9	2	17	13	16
1	-9	2	142	140	6
2	-9	2	214	222	4
3	-9	2	142	149	6
-3	-8	2	91	88	16
-2	-8	2	82	75	11
-1	-8	2	385	405	12
0	-8	2	177	180	9
1	-8	2	242	235	7
2	-8	2	119	125	4
3	-8	2	33	34	32
4	-8	2	93	99	8
-4	-7	2	67	65	16
-3	-7	2	49	40	22
-2	-7	2	129	131	6
-1	-7	2	79	80	7
0	-7	2	437	416	10
1	-7	2	255	254	7
2	-7	2	427	435	6
3	-7	2	182	193	6
4	-7	2	0	20	1
-4	-6	2	69	51	19
-3	-6	2	211	214	6
-2	-6	2	277	284	7
-1	-6	2	433	425	8
0	-6	2	45	44	10
1	-6	2	120	109	6
2	-6	2	201	199	6
3	-6	2	13	19	13
4	-6	2	137	151	6
-4	-5	2	61	60	17
-3	-5	2	44	43	22
-2	-5	2	171	177	5
-1	-5	2	173	183	4
0	-5	2	387	392	7
1	-5	2	584	544	13
2	-5	2	192	187	5
3	-5	2	377	376	7
4	-5	2	0	32	1
-4	-4	2	179	183	7
-3	-4	2	122	123	6
-2	-4	2	225	229	5
-1	-4	2	374	376	7
0	-4	2	129	127	3
1	-4	2	126	124	3
2	-4	2	408	395	8
3	-4	2	114	122	5
4	-4	2	199	206	5
5	-4	2	116	108	15
-4	-3	2	34	35	33
-3	-3	2	153	156	6
-2	-3	2	255	256	5
-1	-3	2	300	316	6
0	-3	2	546	567	12
1	-3	2	611	574	7
2	-3	2	96	102	5
3	-3	2	254	255	5
4	-3	2	34	29	33
5	-3	2	92	82	20
-4	-2	2	113	117	7
-3	-2	2	172	171	5
-2	-2	2	38	24	14
-1	-2	2	476	493	8
0	-2	2	176	170	4
1	-2	2	213	206	5
2	-2	2	574	557	10
3	-2	2	378	375	7
4	-2	2	168	175	6
5	-2	2	150	147	8
-5	-1	2	0	5	1
-4	-1	2	84	76	11
-3	-1	2	139	135	5
-2	-1	2	316	348	26
-1	-1	2	933	1069	18
0	-1	2	111	116	3
1	-1	2	1326	1369	24

Table 6. Observed and calculated structure factors for af012

h	k	l	10Fo	10Fc	10s
2	-1	2	199	186	4
3	-1	2	53	46	12
4	-1	2	63	52	14
5	-1	2	111	113	9
-5	0	2	143	152	8
-4	0	2	75	65	12
-3	0	2	208	206	6
-2	0	2	22	5	21
-1	0	2	528	534	11
0	0	2	89	89	5
1	0	2	422	414	8
2	0	2	647	641	12
3	0	2	345	332	8
4	0	2	139	141	7
5	0	2	94	96	10
-5	1	2	111	115	9
-4	1	2	151	154	7
-3	1	2	220	215	6
-2	1	2	358	366	8
-1	1	2	917	927	20
0	1	2	66	69	5
1	1	2	574	556	10
2	1	2	112	112	6
3	1	2	225	217	6
4	1	2	194	203	7
-5	2	2	116	119	8
-4	2	2	47	42	21
-3	2	2	59	56	14
-2	2	2	316	310	7
-1	2	2	308	294	6
0	2	2	817	892	17
1	2	2	383	387	8
2	2	2	479	472	11
3	2	2	230	232	6
4	2	2	45	44	21
-5	3	2	134	132	13
-4	3	2	233	248	7
-3	3	2	323	318	8
-2	3	2	68	70	7
-1	3	2	298	294	4
0	3	2	250	252	5
1	3	2	113	113	4
2	3	2	269	275	5
3	3	2	135	139	6
-5	4	2	5	15	5
-4	4	2	21	22	21
-3	4	2	152	150	6
-2	4	2	173	179	5
-1	4	2	621	619	13
0	4	2	284	296	5
1	4	2	509	518	11
2	4	2	161	173	5
3	4	2	167	171	6
4	4	2	0	1	1
-5	5	2	179	163	13
-4	5	2	130	123	7
-3	5	2	243	245	5
-2	5	2	206	205	6
-1	5	2	115	102	5
0	5	2	234	230	5
1	5	2	63	64	5
2	5	2	283	267	7
3	5	2	249	249	7
4	5	2	154	158	11
-4	6	2	191	203	9
-3	6	2	104	112	6
-2	6	2	307	313	7
-1	6	2	350	360	8
0	6	2	240	245	5
1	6	2	224	227	6
2	6	2	166	163	6
3	6	2	53	49	15
4	6	2	83	81	19
-4	7	2	80	76	16
-3	7	2	75	78	8
-2	7	2	19	16	18
-1	7	2	20	17	19
0	7	2	263	264	5
1	7	2	244	245	6
2	7	2	316	315	8
3	7	2	164	168	6
4	7	2	133	137	12
-4	8	2	133	149	9
-3	8	2	207	214	6
-2	8	2	241	250	4
-1	8	2	257	273	7
0	8	2	238	236	6
1	8	2	82	72	9
2	8	2	236	242	7
3	8	2	0	27	1
4	8	2	65	62	27
-4	9	2	81	79	18
-3	9	2	22	21	21
-2	9	2	168	177	4
-1	9	2	219	229	6
0	9	2	230	234	9
1	9	2	229	221	9
2	9	2	104	103	11
3	9	2	53	40	31
-3	10	2	174	182	7
-2	10	2	178	189	4
-1	10	2	39	45	28
0	10	2	85	90	15
1	10	2	70	56	20
2	10	2	56	46	21
3	10	2	150	143	10
-3	11	2	83	77	12
-2	11	2	121	126	4
-1	11	2	207	206	6
0	11	2	171	163	9
1	11	2	118	106	11
2	11	2	90	78	14
-2	12	2	67	69	8
-1	12	2	0	5	1
0	12	2	85	79	17
1	12	2	48	41	47
-2	13	2	122	105	11
-1	13	2	137	128	11
0	13	2	141	119	11
1	13	2	97	66	18
-1	-13	3	22	32	22
1	-13	3	114	95	14
2	-13	3	0	13	1
-2	-12	3	20	6	19
-1	-12	3	48	35	48
0	-12	3	103	109	11
1	-12	3	181	157	7
2	-12	3	136	140	4
-1	-11	3	181	183	9
0	-11	3	55	44	28
1	-11	3	76	59	13
2	-11	3	49	55	10
3	-11	3	91	89	10
-3	-10	3	20	18	19
-2	-10	3	163	174	9
-1	-10	3	120	100	10
0	-10	3	281	283	10
1	-10	3	218	215	7
2	-10	3	204	213	4
3	-10	3	20	20	20
-3	-9	3	161	151	10
-2	-9	3	128	137	9
-1	-9	3	48	35	36
0	-9	3	56	46	17
1	-9	3	147	141	6
2	-9	3	100	102	4
3	-9	3	176	184	6
-3	-8	3	87	74	14
-2	-8	3	292	299	10
-1	-8	3	254	262	9
0	-8	3	242	220	7
1	-8	3	271	264	7
2	-8	3	265	264	4
3	-8	3	82	83	9
4	-8	3	16	15	16
-4	-7	3	94	91	11
-3	-7	3	127	124	7
-2	-7	3	151	150	6
-1	-7	3	221	225	6
0	-7	3	111	101	6
1	-7	3	383	363	9
2	-7	3	242	245	4
3	-7	3	114	112	7
4	-7	3	185	194	7
-4	-6	3	90	85	13
-3	-6	3	192	197	6
-2	-6	3	332	346	8
-1	-6	3	259	263	5
0	-6	3	327	318	7
1	-6	3	22	21	22
2	-6	3	117	115	6
3	-6	3	90	87	8
4	-6	3	128	139	7
-4	-5	3	81	68	13
-3	-5	3	97	95	7
-2	-5	3	92	94	5
-1	-5	3	150	155	4
0	-5	3	355	363	7
1	-5	3	547	500	12
2	-5	3	300	298	6
3	-5	3	256	252	6
4	-5	3	164	173	6
-4	-4	3	165	162	7
-3	-4	3	236	237	7
-2	-4	3	523	531	9
-1	-4	3	199	210	4
0	-4	3	257	264	5
1	-4	3	46	36	6
2	-4	3	181	179	4
3	-4	3	111	110	5
4	-4	3	188	200	5
-4	-3	3	45	41	24
-3	-3	3	108	112	7
-2	-3	3	109	118	4
-1	-3	3	583	615	10
0	-3	3	473	474	10
1	-3	3	487	478	6
2	-3	3	636	612	14
3	-3	3	66	59	10
4	-3	3	35	33	34
5	-3	3	0	14	1
-4	-2	3	176	180	7
-3	-2	3	149	147	5
-2	-2	3	638	630	11
-1	-2	3	299	334	7
0	-2	3	274	276	6
1	-2	3	532	505	9
2	-2	3	458	436	8
3	-2	3	226	228	5
4	-2	3	143	144	11
5	-2	3	98	84	17
-5	-1	3	55	56	32
-4	-1	3	38	37	37
-3	-1	3	185	185	8
-2	-1	3	660	627	12
-1	-1	3	819	844	15
0	-1	3	729	768	15
1	-1	3	71	56	5
2	-1	3	50	31	10
3	-1	3	187	183	5
4	-1	3	16	26	16
5	-1	3	81	56	25
-5	0	3	155	162	7
-4	0	3	195	195	5
-3	0	3	201	196	5
-2	0	3	178	157	5
-1	0	3	76	69	5
0	0	3	122	121	6
1	0	3	834	854	15
2	0	3	171	173	4
3	0	3	288	283	6
4	0	3	187	190	7
5	0	3	90	88	20
-5	1	3	73	74	16
-4	1	3	65	54	11
-3	1	3	384	377	9
-2	1	3	304	305	7
-1	1	3	459	441	10
0	1	3	555	579	12
1	1	3	586	593	10
2	1	3	268	253	5
3	1	3	21	5	20
4	1	3	108	107	7
-5	2	3	92	82	13
-4	2	3	111	104	8
-3	2	3	102	96	7
-2	2	3	194	165	6
-1	2	3	109	105	5
1	2	3	556	562	12
2	2	3	459	448	10
3	2	3	129	128	6
4	2	3	81	84	10
-5	3	3	94	94	9
-4	3	3	123	121	7
-3	3	3	169	168	6
-2	3	3	495	484	11
-1	3	3	475	457	6
0	3	3	273	278	6
1	3	3	72	68	6
2	3	3	72	79	9
3	3	3	94	98	7
4	3	3	159	165	10
-5	4	3	81	65	15
-4	4	3	81	76	11
-3	4	3	32	36	32
-2	4	3	214	207	5
-1	4	3	576	560	12
0	4	3	343	354	6
1	4	3	443	464	10
2	4	3	297	302	7
3	4	3	221	219	6
4	4	3	24	31	24
-5	5	3	212	204	12
-4	5	3	132	133	7
-3	5	3	305	300	8
-2	5	3	142	145	5
-1	5	3	81	87	6
0	5	3	116	111	4
1	5	3	83	94	6
2	5	3	293	288	7
3	5	3	175	176	6
4	5	3	114	114	12
-4	6	3	50	45	23
-3	6	3	99	105	10
-2	6	3	229	232	6
-1	6	3	320	322	7
0	6	3	297	307	6
1	6	3	412	415	9
2	6	3	94	93	7
3	6	3	41	26	40
4	6	3	0	11	1
-4	7	3	48	52	33
-3	7	3	180	196	8
-2	7	3	74	77	5
-1	7	3	12	10	12
0	7	3	153	151	4
1	7	3	212	231	6
2	7	3	234	226	7
3	7	3	117	119	7
4	7	3	67	65	15
-4	8	3	115	124	10
-3	8	3	112	117	6
-2	8	3	255	272	4
-1	8	3	247	255	6
0	8	3	116	114	6
1	8	3	108	104	7
2	8	3	15	16	15
3	8	3	127	131	7
4	8	3	99	102	15
-4	9	3	43	40	43
-3	9	3	14	18	13
-2	9	3	96	101	4
-1	9	3	30	20	30
0	9	3	120	126	10
1	9	3	80	76	16
2	9	3	150	154	9
3	9	3	209	203	7
-3	10	3	95	106	12
-2	10	3	40	36	13
-1	10	3	234	233	7
0	10	3	141	139	9
1	10	3	51	35	38
2	10	3	15	10	15
3	10	3	88	85	17
-3	11	3	0	5	1
-2	11	3	36	33	16
-1	11	3	137	128	10
0	11	3	150	145	10
1	11	3	149	131	10
2	11	3	86	73	19
-2	12	3	38	34	15
-1	12	3	86	65	17
0	12	3	71	63	21
1	12	3	42	32	41
-1	13	3	101	92	14
0	13	3	97	84	13
-1	-13	4	119	102	13
0	-13	4	205	189	10
1	-13	4	48	46	47
2	-13	4	40	40	39
-2	-12	4	76	61	25
-1	-12	4	0	19	1
0	-12	4	117	105	11
1	-12	4	197	171	7
2	-12	4	67	65	8
-2	-11	4	193	173	10
-1	-11	4	109	110	10
0	-11	4	120	121	10
1	-11	4	48	48	21
2	-11	4	16	16	16
3	-11	4	57	52	19
-3	-10	4	0	3	1
-2	-10	4	105	98	11
-1	-10	4	56	40	31
0	-10	4	209	199	6
1	-10	4	152	143	6
2	-10	4	149	152	4
3	-10	4	155	158	7
-3	-9	4	96	87	12
-2	-9	4	79	78	12
-1	-9	4	135	117	10
0	-9	4	207	191	6
1	-9	4	61	53	15
2	-9	4	71	63	6
3	-9	4	111	121	7
-3	-8	4	30	12	30
-2	-8	4	126	136	9
-1	-8	4	75	58	11
0	-8	4	395	385	9
1	-8	4	477	443	11
2	-8	4	87	85	5
3	-8	4	91	96	8
4	-8	4	79	71	12
-4	-7	4	115	111	9
-3	-7	4	140	140	7
-2	-7	4	177	182	6
-1	-7	4	48	40	17
0	-7	4	134	128	6
1	-7	4	323	283	8
2	-7	4	53	51	9
3	-7	4	179	178	6
4	-7	4	157	169	7
-4	-6	4	39	29	39
-3	-6	4	188	190	6
-2	-6	4	224	228	6
-1	-6	4	215	223	5
0	-6	4	278	271	7
1	-6	4	384	364	9
2	-6	4	341	347	8
3	-6	4	35	29	35
4	-6	4	29	23	28
-4	-5	4	108	100	8
-3	-5	4	113	116	7
-2	-5	4	23	10	23
-1	-5	4	48	37	10
0	-5	4	214	200	6
1	-5	4	288	269	6
2	-5	4	373	366	7
3	-5	4	333	336	8
4	-5	4	64	53	13
-4	-4	4	153	154	7
-3	-4	4	190	197	6
-2	-4	4	323	333	6
-1	-4	4	242	261	6
0	-4	4	245	244	6
1	-4	4	213	204	3
2	-4	4	174	164	4
3	-4	4	106	112	5
4	-4	4	77	77	7
-4	-3	4	31	22	30
-3	-3	4	44	31	18
-2	-3	4	14	7	13
-1	-3	4	287	293	5
0	-3	4	210	211	5
1	-3	4	551	512	6
2	-3	4	254	256	5
3	-3	4	287	293	6
4	-3	4	133	138	5
5	-3	4	86	77	23
-5	-2	4	65	64	26
-4	-2	4	134	133	7
-3	-2	4	263	261	6
-2	-2	4	383	539	42
-1	-2	4	37	34	11
0	-2	4	113	105	5
1	-2	4	108	93	4
2	-2	4	156	139	4
3	-2	4	92	99	5
4	-2	4	194	188	11
5	-2	4	148	142	13
-5	-1	4	36	36	36
-4	-1	4	27	26	27
-3	-1	4	205	200	5
-2	-1	4	362	350	7
-1	-1	4	89	70	4

Table 6. Observed and calculated structure factors for af012

h	k	l	10Fo	10Fc	10s
0	-1	4	323	330	9
1	-1	4	263	251	5
2	-1	4	584	569	10
3	-1	4	295	289	6
4	-1	4	91	88	8
5	-1	4	30	33	29
-5	0	4	87	82	12
-4	0	4	167	166	5
-3	0	4	154	149	5
-2	0	4	141	141	4
-1	0	4	661	639	12
0	0	4	353	363	12
1	0	4	441	430	8
2	0	4	46	48	11
3	0	4	400	399	9
4	0	4	187	188	7
-5	1	4	43	34	43
-4	1	4	0	20	1
-3	1	4	213	210	6
-2	1	4	239	229	6
-1	1	4	888	856	16
0	1	4	488	535	10
1	1	4	470	474	8
2	1	4	92	92	4
3	1	4	181	179	6
4	1	4	81	64	11
-5	2	4	117	117	8
-4	2	4	181	168	7
-3	2	4	264	242	7
-2	2	4	86	78	7
-1	2	4	49	39	15
0	2	4	374	378	8
1	2	4	398	407	7
2	2	4	149	153	4
3	2	4	280	283	7
4	2	4	167	172	7
-5	3	4	38	15	38
-4	3	4	122	122	7
-3	3	4	263	257	7
-2	3	4	487	484	11
-1	3	4	364	359	5
0	3	4	385	403	11
1	3	4	100	106	5
2	3	4	201	197	6
3	3	4	51	42	18
4	3	4	93	91	9
-5	4	4	157	158	8
-4	4	4	100	93	8
-3	4	4	57	49	15
-2	4	4	203	190	6
-1	4	4	108	109	7
0	4	4	297	310	7
1	4	4	427	433	9
2	4	4	313	319	7
3	4	4	122	126	7
4	4	4	100	101	8
-5	5	4	104	83	22
-4	5	4	116	117	7
-3	5	4	263	255	7
-2	5	4	220	220	8
-1	5	4	244	244	6
0	5	4	332	319	8
1	5	4	42	25	17
2	5	4	120	116	6
3	5	4	30	23	29
4	5	4	101	102	8
-4	6	4	158	148	7
-3	6	4	49	49	27
-2	6	4	116	115	6
-1	6	4	313	314	7
0	6	4	245	253	5
1	6	4	394	399	9
2	6	4	225	226	6
3	6	4	126	126	7
4	6	4	38	41	38
-4	7	4	101	105	11
-3	7	4	239	262	9
-2	7	4	199	206	3
-1	7	4	147	148	5
0	7	4	27	25	27
1	7	4	70	60	10
2	7	4	145	139	6
3	7	4	167	162	7
4	7	4	64	53	29
-4	8	4	19	21	18
-3	8	4	75	74	16
-2	8	4	197	207	3
-1	8	4	244	251	6
0	8	4	327	322	8
1	8	4	103	106	7
2	8	4	232	225	7
3	8	4	93	87	9
-4	9	4	115	121	11
-3	9	4	79	80	12
-2	9	4	92	94	4
-1	9	4	170	177	6
0	9	4	243	243	9
1	9	4	81	73	13
2	9	4	254	248	7
3	9	4	157	150	7
-3	10	4	110	106	11
-2	10	4	210	218	4
-1	10	4	241	250	7
0	10	4	183	178	9
1	10	4	88	72	16
2	10	4	66	71	17
-3	11	4	91	97	14
-2	11	4	119	128	4
-1	11	4	0	11	1
0	11	4	138	135	10
1	11	4	146	126	10
2	11	4	127	116	11
-2	12	4	154	159	5
-1	12	4	106	92	12
0	12	4	106	100	12
1	12	4	25	9	25
-1	13	4	100	86	18
0	13	4	124	109	11
0	-13	5	137	119	11
1	-13	5	70	67	26
-2	-12	5	25	42	24
-1	-12	5	0	15	1
0	-12	5	44	26	43
1	-12	5	20	13	20
2	-12	5	78	80	7
-2	-11	5	143	131	11
-1	-11	5	140	152	10
0	-11	5	151	154	9
1	-11	5	207	183	7
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Table 6. Observed and calculated structure factors for af012

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3	-1	8	317	302	8
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3	0	8	307	302	8
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2	1	8	207	212	6
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Table 6. Observed and calculated structure factors for af012

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3	4	8	192	193	6
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2	5	8	165	170	6
3	5	8	136	135	7
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0	-11	11	22	20	21
1	-11	11	18	12	18
2	-11	11	89	77	13
-1	-10	11	146	141	7
0	-10	11	247	243	7
1	-10	11	158	156	4
2	-10	11	160	156	9
3	-10	11	72	70	25
-1	-9	11	49	36	20

Table 6. Observed and calculated structure factors for af012

h	k	l	10Fo	10Fc	10s
0	-9	11	45	35	22
1	-9	11	174	176	4
2	-9	11	154	142	7
3	-9	11	35	31	34
-1	-8	11	131	125	6
0	-8	11	228	233	6
1	-8	11	207	207	4
2	-8	11	72	59	13
3	-8	11	61	48	17
-2	-7	11	42	42	42
-1	-7	11	121	126	6
0	-7	11	77	69	9
1	-7	11	228	222	4
2	-7	11	313	308	8
3	-7	11	115	112	8
4	-7	11	151	147	12
-2	-6	11	324	343	12
-1	-6	11	190	183	6
0	-6	11	225	231	6
1	-6	11	106	117	4
2	-6	11	12	8	12
3	-6	11	129	132	7
4	-6	11	38	28	38
-4	-5	11	50	47	50
-3	-5	11	56	55	19
-2	-5	11	19	16	19
-1	-5	11	195	199	10
0	-5	11	304	306	7
1	-5	11	333	325	4
2	-5	11	217	204	6
3	-5	11	107	100	7
4	-5	11	120	119	11
-4	-4	11	120	115	14
-3	-4	11	150	151	7
-2	-4	11	291	314	8
-1	-4	11	275	276	11
0	-4	11	25	21	25
1	-4	11	25	18	25
2	-4	11	93	75	8
3	-4	11	83	70	12
4	-4	11	116	115	11
-4	-3	11	19	34	18
-3	-3	11	0	13	1
-2	-3	11	300	308	12
-1	-3	11	235	246	10
0	-3	11	314	311	7
1	-3	11	264	253	6
2	-3	11	289	306	6
3	-3	11	111	102	6
4	-3	11	82	62	20
-4	-2	11	95	94	9
-3	-2	11	113	101	8
-2	-2	11	243	245	10
-1	-2	11	31	22	30
0	-2	11	134	128	5
1	-2	11	20	27	19
2	-2	11	192	200	7
3	-2	11	271	257	7
4	-2	11	128	120	7
-4	-1	11	58	51	21
-3	-1	11	32	22	32
-2	-1	11	202	199	10
-1	-1	11	354	367	13
0	-1	11	431	435	15
1	-1	11	415	415	8
2	-1	11	241	253	5
3	-1	11	48	46	23
4	-1	11	22	29	21
-4	0	11	111	111	8
-3	0	11	113	117	7
-2	0	11	326	300	12
-1	0	11	60	54	29
0	0	11	270	268	10
1	0	11	415	412	8
2	0	11	211	211	5
3	0	11	125	124	7
4	0	11	200	188	7
-4	1	11	92	81	10
-3	1	11	197	193	10
-2	1	11	392	386	14
-1	1	11	233	245	9
0	1	11	328	341	12
1	1	11	67	54	7
2	1	11	104	104	6
3	1	11	75	64	11
4	1	11	105	96	9
-4	2	11	65	64	27

h	k	l	10Fo	10Fc	10s
-3	2	11	22	8	22
-2	2	11	115	99	10
-1	2	11	24	18	24
0	2	11	193	198	9
1	2	11	452	450	8
2	2	11	203	232	6
3	2	11	221	227	6
4	2	11	56	59	23
-4	3	11	171	176	10
-3	3	11	134	121	10
-2	3	11	201	193	9
-1	3	11	278	272	11
0	3	11	192	150	10
1	3	11	70	62	9
2	3	11	83	110	14
3	3	11	83	81	9
4	3	11	66	55	18
-4	4	11	23	32	23
-3	4	11	29	26	29
-2	4	11	116	100	10
-1	4	11	150	144	8
0	4	11	238	231	7
1	4	11	280	292	7
2	4	11	226	231	6
3	4	11	102	95	8
4	4	11	73	79	19
-4	5	11	87	85	14
-3	5	11	111	106	11
-2	5	11	246	235	10
-1	5	11	101	102	9
0	5	11	102	97	7
1	5	11	81	81	8
2	5	11	116	121	6
3	5	11	134	107	8
4	5	11	118	104	12
-4	6	11	53	50	52
-3	6	11	38	27	37
-2	6	11	287	288	11
-1	6	11	152	150	9
0	6	11	133	125	10
1	6	11	272	284	7
2	6	11	117	113	7
3	6	11	82	80	9
-3	7	11	88	83	12
-2	7	11	16	13	15
-1	7	11	19	22	19
0	7	11	127	129	9
1	7	11	152	189	23
2	7	11	158	149	6
3	7	11	152	152	7
-3	8	11	126	112	11
-2	8	11	84	74	12
0	8	11	119	128	10
1	8	11	150	145	10
2	8	11	0	9	1
3	8	11	38	31	38
-3	9	11	62	62	21
-2	9	11	67	55	16
-1	9	11	80	82	13
0	9	11	110	108	11
1	9	11	104	104	11
2	9	11	106	104	8
-3	10	11	83	92	12
1	10	11	0	17	1
2	10	11	19	23	18
0	-12	12	36	32	36
1	-12	12	104	103	6
0	-11	12	124	123	11
1	-11	12	180	184	4
2	-11	12	34	32	34
-1	-10	12	170	170	10
0	-10	12	126	120	11
1	-10	12	255	260	4
2	-10	12	103	108	8
-1	-9	12	39	36	39
0	-9	12	41	43	41
1	-9	12	45	46	11
2	-9	12	14	16	13
3	-9	12	96	92	11
-2	-8	12	92	93	12
-1	-8	12	75	82	13
0	-8	12	171	192	9
1	-8	12	283	276	4
2	-8	12	186	177	7
3	-8	12	176	178	7
-2	-7	12	73	59	22
-1	-7	12	165	184	9

h	k	l	10Fo	10Fc	10s
0	-7	12	23	24	23
1	-7	12	74	75	5
2	-7	12	179	183	6
3	-7	12	134	142	7
-2	-6	12	136	128	11
-1	-6	12	262	242	11
0	-6	12	288	316	11
1	-6	12	221	220	3
2	-6	12	127	112	7
3	-6	12	114	118	8
4	-6	12	0	8	1
-4	-5	12	91	85	21
-3	-5	12	94	91	10
-2	-5	12	128	114	11
-1	-5	12	0	16	1
0	-5	12	102	110	11
1	-5	12	254	253	6
2	-5	12	165	155	6
3	-5	12	305	303	8
4	-5	12	184	189	10
-4	-4	12	99	97	15
-3	-4	12	227	235	7
-2	-4	12	264	267	11
-1	-4	12	252	273	10
0	-4	12	256	280	11
1	-4	12	267	262	7
2	-4	12	45	44	19
3	-4	12	88	84	8
4	-4	12	123	127	11
-4	-3	12	58	49	39
-3	-3	12	81	82	10
-2	-3	12	178	177	10
-1	-3	12	74	77	19
0	-3	12	295	285	11
1	-3	12	423	412	8
2	-3	12	321	332	6
3	-3	12	143	141	6
4	-3	12	128	131	11
-4	-2	12	78	58	15
-3	-2	12	282	294	8
-2	-2	12	276	279	11
-1	-2	12	230	236	10
0	-2	12	177	163	10
1	-2	12	23	39	23
2	-2	12	161	155	5
3	-2	12	122	116	7
4	-2	12	197	168	10
-4	-1	12	39	38	38
-3	-1	12	24	25	24
-2	-1	12	306	304	12
-1	-1	12	334	344	12
0	-1	12	416	431	15
1	-1	12	329	330	9
2	-1	12	198	196	5
3	-1	12	110	105	8
4	-1	12	41	38	40
-4	0	12	175	179	7
-3	0	12	233	235	10
-2	0	12	208	199	10
-1	0	12	74	55	18
0	0	12	27	5	27
1	0	12	198	193	6
2	0	12	58	53	14
3	0	12	170	163	6
4	0	12	210	204	7
-4	1	12	32	32	31
-3	1	12	200	198	10
-2	1	12	232	215	10
-1	1	12	170	176	9
0	1	12	510	519	17
1	1	12	167	176	6
2	1	12	321	322	8
3	1	12	21	8	21
4	1	12	48	49	30
-4	2	12	79	68	22
-3	2	12	181	171	10
-2	2	12	195	179	10
-1	2	12	36	32	35
0	2	12	217	208	9
1	2	12	284	289	8
2	2	12	283	282	7
3	2	12	156	158	6
4	2	12	116	108	9
-4	3	12	89	53	25
-3	3	12	168	125	13
-2	3	12	476	451	17
-1	3	12	249	240	10

h	k	l	10Fo	10Fc	10s
0	3	12	237	247	7
1	3	12	127	134	10
2	3	12	52	42	16
3	3	12	24	25	24
4	3	12	100	94	13
-4	4	12	83	78	19
-3	4	12	66	54	23
-2	4	12	72	64	14
-1	4	12	198	202	9
0	4	12	300	304	8
1	4	12	265	272	11
2	4	12	261	270	7
3	4	12	135	135	7
4	4	12	68	47	27
-4	5	12	172	162	11
-3	5	12	131	122	11
-2	5	12	387	368	14
-1	5	12	188	184	9
0	5	12	119	117	11
1	5	12	117	119	11
2	5	12	75	70	19
3	5	12	89	65	12
-3	6	12	120	111	11
-2	6	12	212	207	10
-1	6	12	40	24	40
0	6	12	426	443	16
1	6	12	108	105	11
2	6	12	142	144	10
3	6	12	70	67	24
-3	7	12	81	76	18
-2	7	12	121	118	10
-1	7	12	118	121	9
0	7	12	49	40	27
1	7	12	178	183	10
2	7	12	76	75	18
3	7	12	131	124	13
-3	8	12	95	93	11
-2	8	12	182	185	9
0	8	12	165	170	9
1	8	12	143	141	10
2	8	12	51	42	38
-3	9	12	62	57	22
-2	9	12	18	15	17
-1	9	12	21	17	21
0	9	12	33	43	32
1	9	12	149	142	10
2	9	12	52	50	39
1	10	12	15	19	14
2	10	12	69	55	23
0	-12	13	0	12	1
0	-11	13	177	171	11
-1	-10	13	94	108	11
0	-10	13	70	74	21
1	-10	13	168	161	11
2	-10	13	163	158	8
-1	-9	13	170	174	10
0	-9	13	167	168	10
1	-9	13	98	93	12
2	-9	13	33	27	32
3	-9	13	100	102	13
-2	-8	13	72	80	21
-1	-8	13	140	165	9
0	-8	13	267	290	11
1	-8	13	254	256	11
2	-8	13	207	206	7
3	-8	13	186	184	7
-2	-7	13	189	186	10
-1	-7	13	210	230	10
0	-7	13	33	25	32
1	-7	13	49	58	40
2	-7	13	141	142	7
3	-7	13	142	136	7
-2	-6	13	125	128	12
-1	-6	13	266	251	12
0	-6	13	303	358	11
1	-6	13	250	241	11
2	-6	13	192	184	6
3	-6	13	107	101	8
4	-6	13	26	28	26
-3	-5	13	180	185	11
-2	-5	13	134	145	10
-1	-5	13	41	44	41
0	-5	13	92	91	12
1	-5	13	131	134	9
2	-5	13	195	248	24
3	-5	13	258	253	7
4	-5	13	95	74	17

h	k	l	10Fo	10Fc	10s
-4	-4	13	52	44	52
-3	-4	13	176	189	7
-2	-4	13	194	201	10
-1	-4	13	302	330	12
0	-4	13	270	280	11
1	-4	13	199	202	10
2	-4	13	77	66	16
3	-4	13	23	9	22
4	-4	13	34	41	33
-4	-3	13	93	89	11
-3	-3	13	25	21	25
-2	-3	13	22	9	22
-1	-3	13	10	7	10
0	-3	13	231	232	10
1	-3	13	236	243	7
2	-3	13	129	129	7
3	-3	13	296	292	10
4	-3	13	148	139	10
-4	-2	13	102	100	11
-3	-2	13	296	243	13
-2	-2	13	84	84	14
-1	-2	13	309	335	12
0	-2	13	312	307	12
1	-2	13	141	172	6
2	-2	13	58	50	15
3	-2	13	34	32	33
4	-2	13	139	134	11
-4	-1	13	25	30	24
-3	-1	13	73	57	23
-2	-1	13	264	200	13
-1	-1	13	39	25	38
0	-1	13	357	358	13
1	-1	13	418	425	10
2	-1	13	122	126	7
3	-1	13	204	199	11
4	-1	13	96	90	11
-4	0	13	134	128	12
-3	0	13	247	233	11
-2	0	13	156	143	10
-1	0	13	196	198	9
0	0	13	101	104	11
1	0	13	106	100	7
2	0	13	63	64	25
3	0	13	203	205	11
4	0	13	114	106	16
-4	1	13	0	11	1
-3	1	13	93	80	13
-2	1	13	90	88	12
-1	1	13	437	446	15
0	1	13	172	174	9
1	1	13	384	386	10
2	1	13	309	302	13
3	1	13	29	26	28
4	1	13	32	30	31
-4	2	13	143	134	12
-3	2	13	204	199	11
-2	2	13	129	122	9
-1	2	13	36	26	36
0	2	13	104	95	11
1	2	13	104	101	12
2	2	13	141	140	11
3	2	13	182	175	11
4	2	13	92	89	20
-4	3	13	90	78	21
-3	3	13	244	238	11
-2	3	13	87	87	12
-1	3	13	285	275	11
0	3	13	242	244	7
1	3	13	149	152	10
2	3	13	167	164	11
3	3	13	5	21	5
-4	4	13	65	43	33
-3	4	13	170	137	13
-2	4	13	0	6	1
-1	4	13	65	67	14
0	4	13	143	136	6
1	4	13	211	221	10
2	4	13	95	85	13
3	4	13	131	136	11
-3	5	13	326	322	13
-2	5	13	114	107	11
-1	5	13	153	145	9
0	5	13	88	91	12
1	5	13	66	50	20
2	5	13	131	135	11
3	5	13	5	10	5
-3	6	13	86	87	14

Table 6. Observed and calculated structure factors for af012

h	k	l	10Fo	10Fc	10s
-2	6	13	5	16	5
-1	6	13	57	54	20
0	6	13	182	173	10
1	6	13	73	72	17
2	6	13	232	235	11
3	6	13	134	124	13
-3	7	13	79	71	19
-2	7	13	229	226	10
0	7	13	113	118	10
1	7	13	71	62	18
2	7	13	20	8	20
-3	8	13	49	50	48
-2	8	13	75	64	17
0	8	13	172	173	10
1	8	13	118	106	11
2	8	13	42	43	42
-3	9	13	81	65	19
-2	9	13	126	131	10
-1	9	13	27	6	26
0	9	13	40	35	40
1	9	13	30	30	29
2	9	13	50	45	49
0	10	13	52	43	33
1	10	13	123	122	10
0	-11	14	189	181	11
1	-11	14	96	81	19
-1	-10	14	61	55	29
0	-10	14	78	65	20
1	-10	14	86	87	17
-1	-9	14	215	213	11
0	-9	14	142	144	10
1	-9	14	139	132	11
2	-9	14	91	77	20
-2	-8	14	24	14	23
-1	-8	14	114	130	11
0	-8	14	77	80	17
1	-8	14	187	197	10
2	-8	14	194	180	11
3	-8	14	146	125	12
-2	-7	14	142	144	11
-1	-7	14	171	176	10
0	-7	14	96	89	12
1	-7	14	68	58	22
2	-7	14	43	31	43
3	-7	14	88	71	16
-3	-6	14	47	44	47
-2	-6	14	55	51	48
-1	-6	14	214	200	11
0	-6	14	170	196	9
1	-6	14	142	146	10
2	-6	14	257	263	11
3	-6	14	162	147	11
-3	-5	14	225	232	11
-2	-5	14	133	139	11
-1	-5	14	132	134	10
0	-5	14	82	85	13
1	-5	14	21	32	21
2	-5	14	121	128	10
3	-5	14	85	80	15
-3	-4	14	84	78	21
-2	-4	14	114	122	11
-1	-4	14	204	216	10
0	-4	14	290	308	12
1	-4	14	269	278	11
2	-4	14	198	197	10
3	-4	14	147	153	10
-4	-3	14	94	87	24
-3	-3	14	107	99	13
-2	-3	14	257	269	11
-1	-3	14	77	67	24
0	-3	14	39	16	39
1	-3	14	154	155	6
2	-3	14	277	285	11
-4	-2	14	41	24	41
-3	-2	14	96	91	13
-2	-2	14	209	210	10
-1	-2	14	508	524	18
0	-2	14	311	301	12
1	-2	14	198	211	6
2	-2	14	0	16	1
3	-2	14	35	31	34
-4	-1	14	130	126	14
-3	-1	14	101	99	13
-2	-1	14	42	35	42
-1	-1	14	75	74	19
0	-1	14	415	423	15
1	-1	14	65	57	15
2	-1	14	299	304	8
3	-1	14	215	214	11
4	-1	14	112	107	16
-4	0	14	172	167	12
-2	0	14	190	184	10
-1	0	14	368	358	14
0	0	14	108	105	11
1	0	14	336	345	9
2	0	14	104	96	13
3	0	14	79	63	21
4	0	14	124	112	15
-4	1	14	23	21	23
-3	1	14	52	46	41
-2	1	14	60	55	24
-1	1	14	76	66	19
0	1	14	389	390	14
1	1	14	333	340	9
2	1	14	163	162	11
3	1	14	144	148	11
4	1	14	40	45	39
-4	2	14	118	120	13
-3	2	14	157	150	10
-2	2	14	106	103	11
-1	2	14	336	336	12
0	2	14	72	58	18
1	2	14	49	43	41
2	2	14	123	118	12
3	2	14	85	74	18
-4	3	14	36	30	36
-3	3	14	28	24	28
-2	3	14	154	149	9
-1	3	14	148	144	9
0	3	14	372	373	10
1	3	14	290	299	12
2	3	14	159	144	11
3	3	14	104	109	13
-3	4	14	304	294	12
-2	4	14	39	29	39
-1	4	14	165	158	9
0	4	14	39	36	39
1	4	14	74	73	18
2	4	14	163	164	11
3	4	14	165	163	11
-3	5	14	92	74	25
-2	5	14	113	108	10
-1	5	14	257	250	10
0	5	14	139	132	10
1	5	14	350	385	14
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Table 6. Observed and calculated structure factors for af012

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2	0	17	204	205	11
3	0	17	108	114	14
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2	3	20	32	26	32
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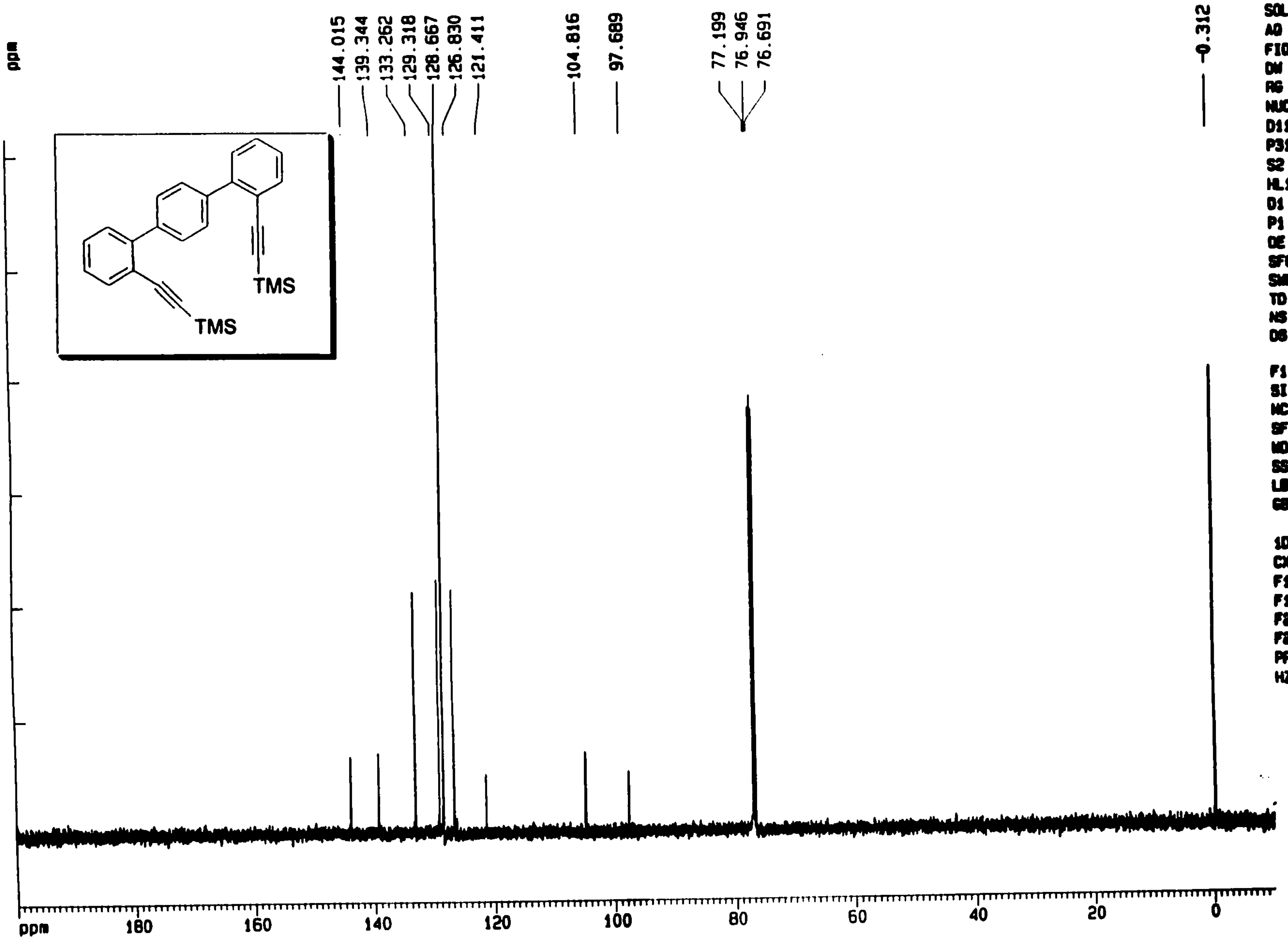
-1	4	20	87	91	15
0	4	20	81	64	20
1	4	20	141	131	12
-2	5	20	81	71	21
0	5	20	38	31	38
1	5	20	67	47	29
-2	6	20	21	31	20
0	6	20	153	148	12
0	-6	21	146	153	11
1	-6	21	148	153	11
-1	-5	21	63	42	62
0	-5	21	71	61	26
1	-5	21	72	62	26
2	-5	21	117	110	15
-1	-4	21	155	161	11
0	-4	21	183	190	11
1	-4	21	200	193	11
2	-4	21	32	46	31
-2	-3	21	33	35	33
-1	-3	21	99	94	15
0	-3	21	93	90	15
1	-3	21	147	154	11
-2	-2	21	49	48	49
-1	-2	21	152	148	11
0	-2	21	102	92	14
1	-2	21	58	42	37
-2	-1	21	32	29	32
-1	-1	21	130	128	12
0	-1	21	109	96	9
1	-1	21	198	185	12
-2	0	21	53	41	53
-1	0	21	130	138	12
0	0	21	74	57	18
1	0	21	14	3	13
2	0	21	57	56	57
-2	1	21	17	20	16
-1	1	21	144	143	12
0	1	21	188	170	8
1	1	21	93	100	16
2	1	21	116	118	16
-2	2	21	88	77	23
-1	2	21	16	33	15
0	2	21	27	28	26
1	2	21	19	14	18
2	2	21	105	87	21
-2	3	21	80	79	26
-1	3	21	102	96	12
0	3	21	172	163	12
-2	4	21	0	5	1
0	4	21	94	75	16
1	4	21	129	126	13
-2	5	21	127	100	19
0	5	21	117	110	14
0	-5	22	87	69	22
1	-5	22	58	56	44
-1	-4	22	117	115	15
0	-4	22	225	231	11
1	-4	22	102	97	20
-1	-3	22	69	50	36
0	-3	22	68	54	29
1	-3	22	26	24	26
-1	-2	22	180	186	11
0	-2	22	241	206	13
1	-2	22	165	167	11
-1	-1	22	34	24	33
0	-1	22	163	164	8
-2	0	22	162	166	12
-1	0	22	201	186	12
0	0	22	106	96	10
1	0	22	0	9	1
2	0	22	60	55	59
-2	1	22	92	99	16
-1	1	22	99	100	14
0	1	22	160	146	8
1	1	22	122	117	15
-2	2	22	89	73	22
-1	2	22	90	72	20
0	2	22	95	86	15
1	2	22	59	49	45
-1	3	22	109	112	13
0	3	22	203	190	12
1	3	22	87	79	23
0	4	22	28	11	27
0	-3	23	74	58	27
1	-3	23	66	58	35
-1	-2	23	147	146	14
0	-2	23	199	191	11
1	-2	23	103	101	15
-1	-1	23	92	93	16
0	-1	23	35	29	34
-1	0	23	93	76	20
0	0	23	94	79	14
1	0	23	118	108	15
-1	1	23	87	65	23
0	1	23	15	19	15
1	1	23	182	166	13
-1	2	23	108	119	14
0	2	23	87	58	22
0	-2	24	128	118	13
0	-1	24	15	33	14
0	0	24	145	143	9
1	0	24	162	153	13
0	1	24	42	39	42
-1	2	24	130	123	13

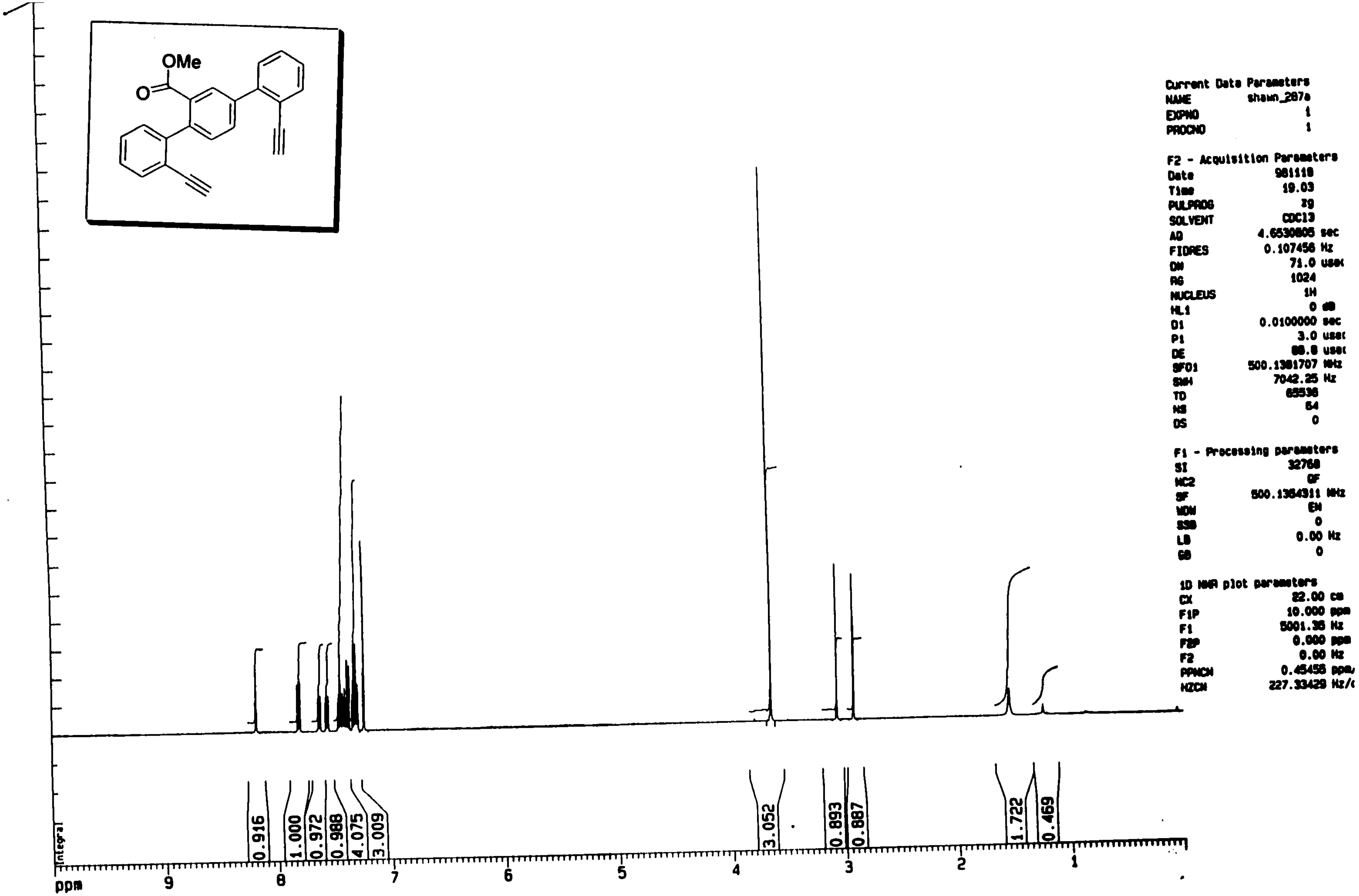


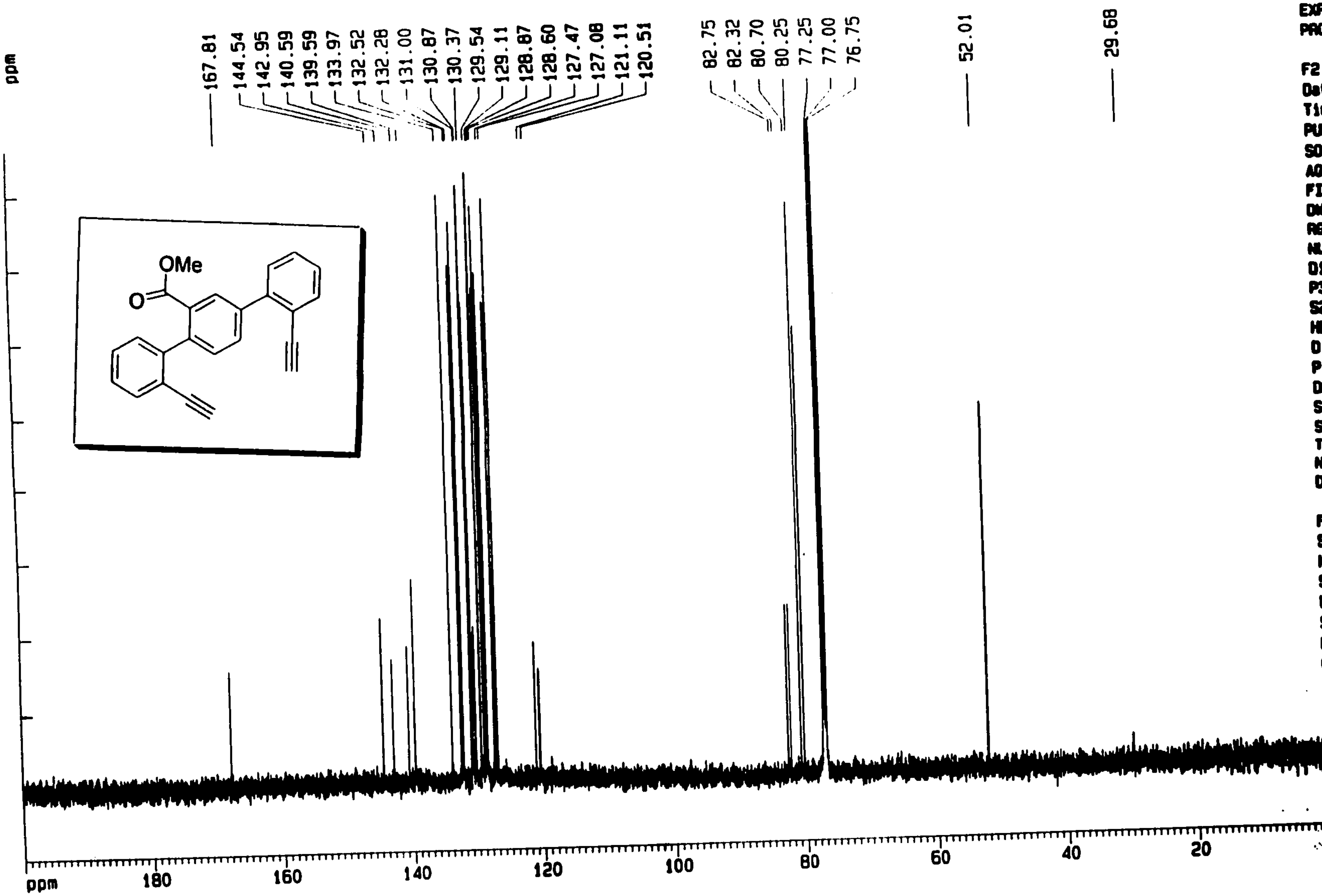
```
Current Data Parameters
NAME           shawn_319a
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parame
Date               990128
Time                 8.35
PULPROG              zgdc
SOLVENT             CDCl3
AQ              1.0485960
FIDRES           0.476837
DW                   16.0
RG                  32768
NUCLEUS               13C
D11             0.0300000
P31                  70.0
S2                     22
HL1                    22
D1              1.0000000
P1                    5.0
DE                   20.0
SFO1          125.7724464
SWH              31250.00
TD                  65536
NS                   1185
DS                      0

F1 - Processing paramet
SI                  32768
MC2                    QF
SF            125.7581571
WDW                    EM
SSB                     0
LB                   1.00
GB                      0

1D NMR plot parameters
CX                  22.00
F1P               200.000
F1               25151.83
F2P               -10.000
F2               -1257.59
PPMCM             9.54545
HZCM           1200.42834
```


OMe
O
Current Data Parameters
NAME shawn_287a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 981118
Time 19.03
PULPROG zg
SOLVENT CDCl3
AQ 4.6530805 sec
FIDRES 0.107456 Hz
DW 71.0 usec
RG 1024
NUCLEUS 1H
HL1 0 dB
D1 0.0100000 sec
P1 3.0 usec
DE 88.8 usec
SFO1 500.1381707 MHz
SWH 7042.25 Hz
TD 65536
NS 64
DS 0
F1 - Processing parameters
SI 32768
MC2 QF
SF 500.1364311 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 22.00 cm
F1P 10.000 ppm
F1 5001.36 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.45455 ppm/
HZCM 227.33429 Hz/c
Integral
0.916
1.000
0.972
0.988
4.075
3.009
3.052
0.893
0.887
1.722
0.469
ppm
9
8
7
6
5
4
3
2
1

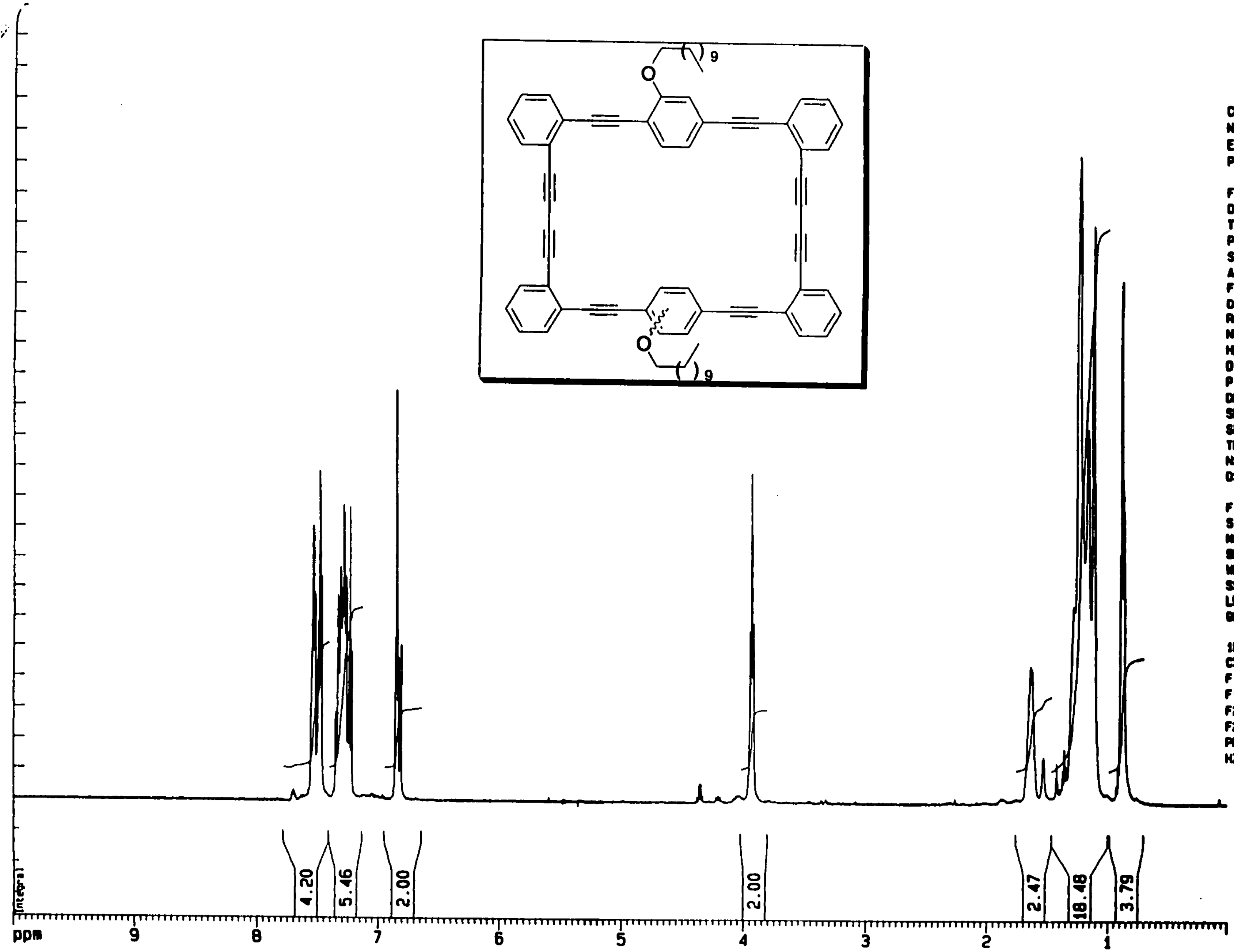


```
Current Data Parameters
NAME             shawn_287a
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parame
Date                 981118
Time                  19.42
PULPROG                zgdc
SOLVENT               CDCl3
AQ                1.0485960
FIDRES             0.476837
DW                     16.0
RG                    32768
NUCLEUS                 13C
D11               0.0300000
P31                    70.0
S2                       22
HL1                      22
D1                1.0000000
P1                      5.0
DE                     20.0
SFO1            125.7724464
SWH                31250.00
TD                    65536
NS                    18432
DS                        0

F1 - Processing paramet
SI                    32768
MC2                      QF
SF              125.7591504
WDW                      EM
SSB                       0
LB                     1.00
GB                        0

1D NMR plot parameters
CX                    22.00
F1P                 200.000
F1                 25151.63
F2P                   0.000
F2                     0.00
PPMCM               9.09091
HZCM             1143.28501
```

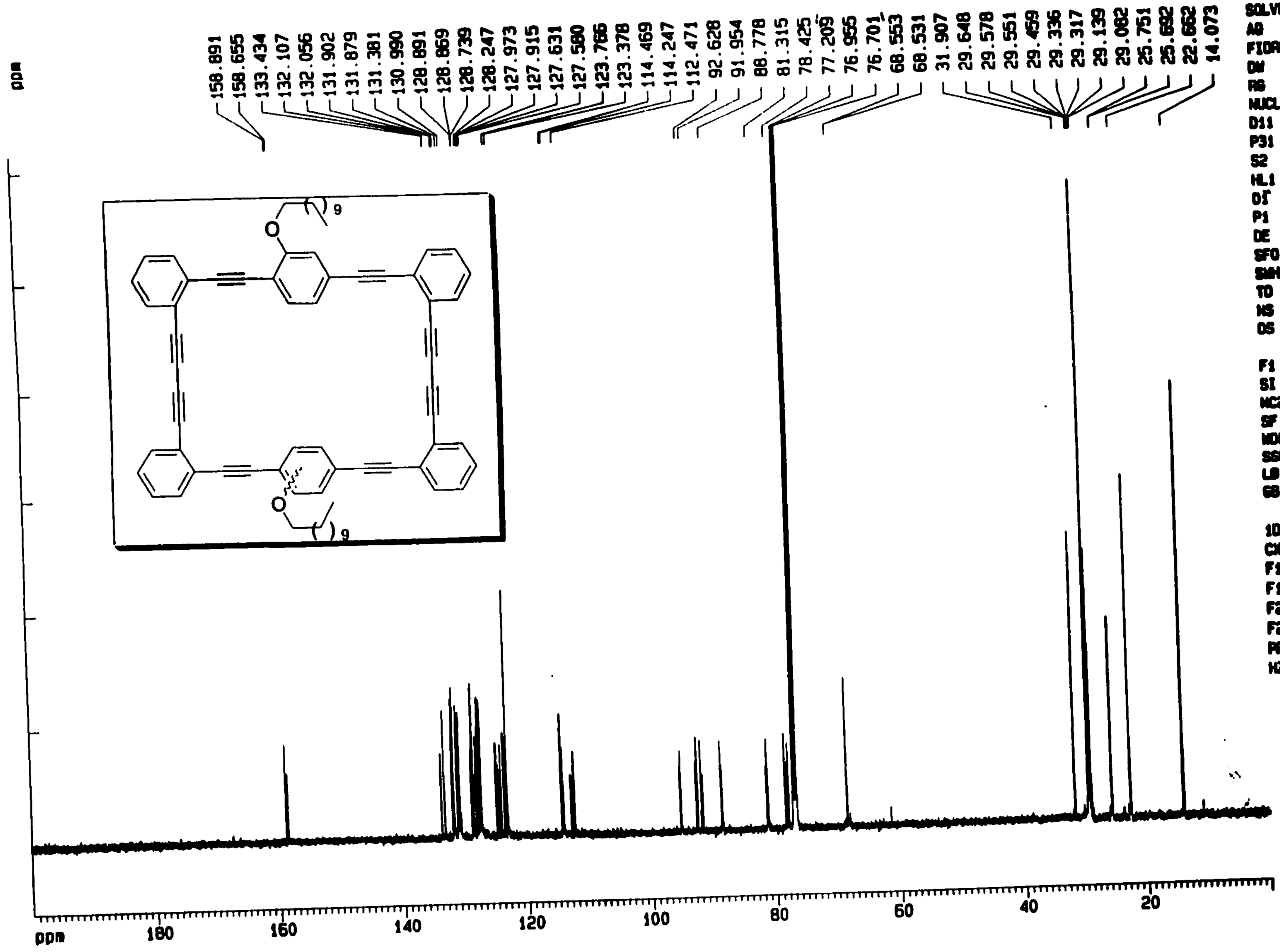


```
Current Data Parameters
NAME          shawn_343a
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date              981102
Time               20.41
PULPROG               zg
SOLVENT            CDCl3
AQ          4.6530805 sec
FIDRES       0.107456 Hz
DW              71.0 use
RG                   128
NUCLEUS               1H
HL1                 0 dB
D1         0.0100000 sec
P1               3.0 use
DE              88.8 use
SFO1     500.1381707 MHz
SWH           7042.25 Hz
TD                 65536
NS                    16
DS                     0

F1 - Processing parameters
SI                 32768
MC2                   QF
SF       500.1354311 MHz
WDW                   EM
SSB                    0
LB               0.00 Hz
GB                     0

1D NMR plot parameters
CX              22.00 cm
F1P           10.000 ppm
F1            5001.35 Hz
F2P            0.000 ppm
F2               0.00 Hz
PPMCM        0.45455 ppm/
HZCM       227.33429 Hz/
```



```
Current Data Parameters
NAME           shawn_343a
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parame
Date               991102
Time                20.46
PULPROG              zgdc
SOLVENT             CDCl3
AQ              1.0485860
FIDRES           0.476837
DW                   16.0
RG                  32768
NUCLEUS               13C
D11             0.0300000
P31                  70.0
S2                     22
HL1                    22
D1              5.0000000
P1                    5.0
DE                   20.0
SFO1          125.7724464
SWH              31250.00
TD                  65536
NS                   4608
DS                      0

F1 - Processing paramet
SI                  32768
MC2                    QF
SF            125.7591571
WDW                    EM
SSB                     0
LB                   1.00
GB                      0

1D NMR plot parameters
CX                  22.00
F1P               200.000
F1               25151.83
F2P                 0.000
F2                   0.00
PPMCM             9.09091
HZCM           1143.26501
```

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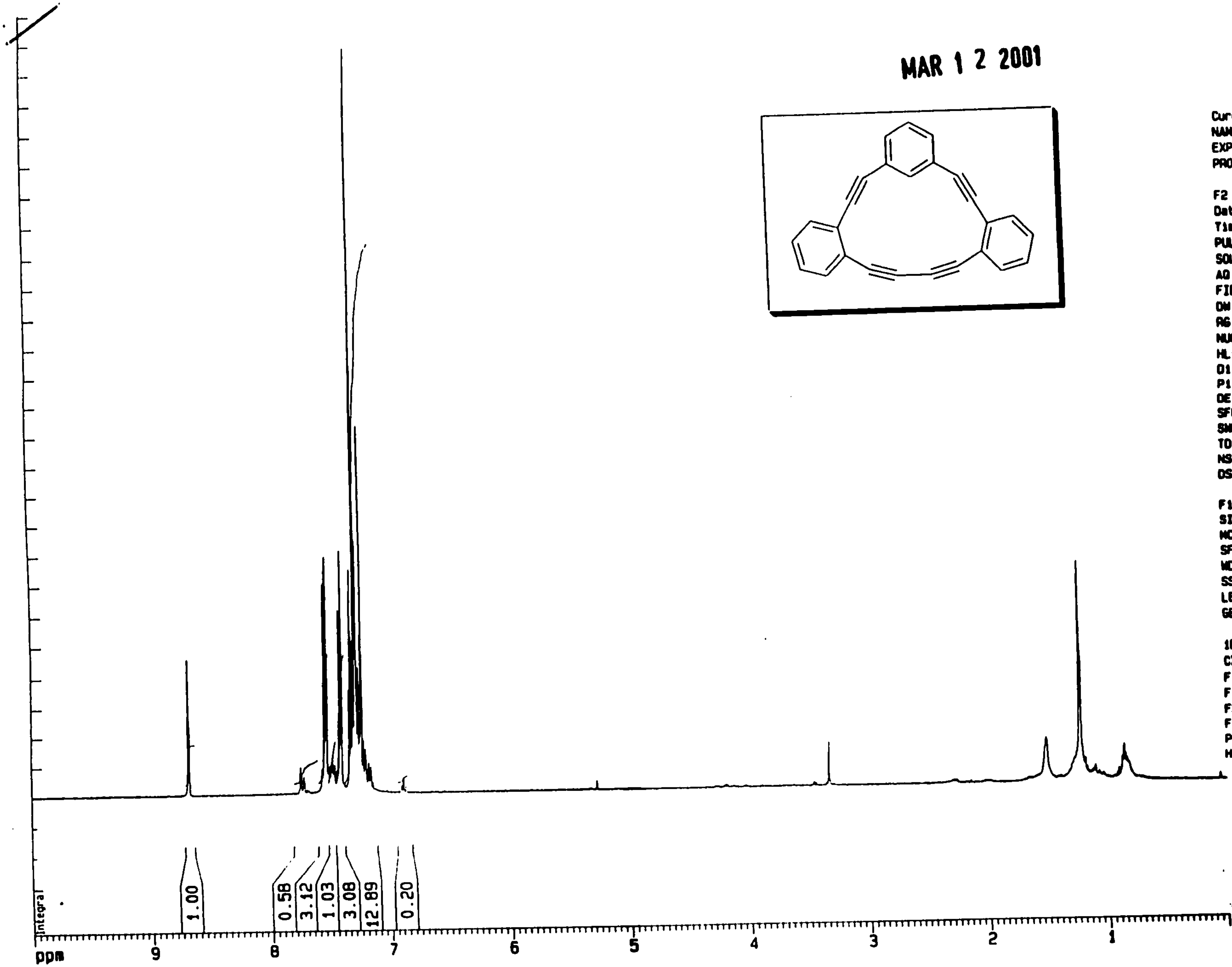
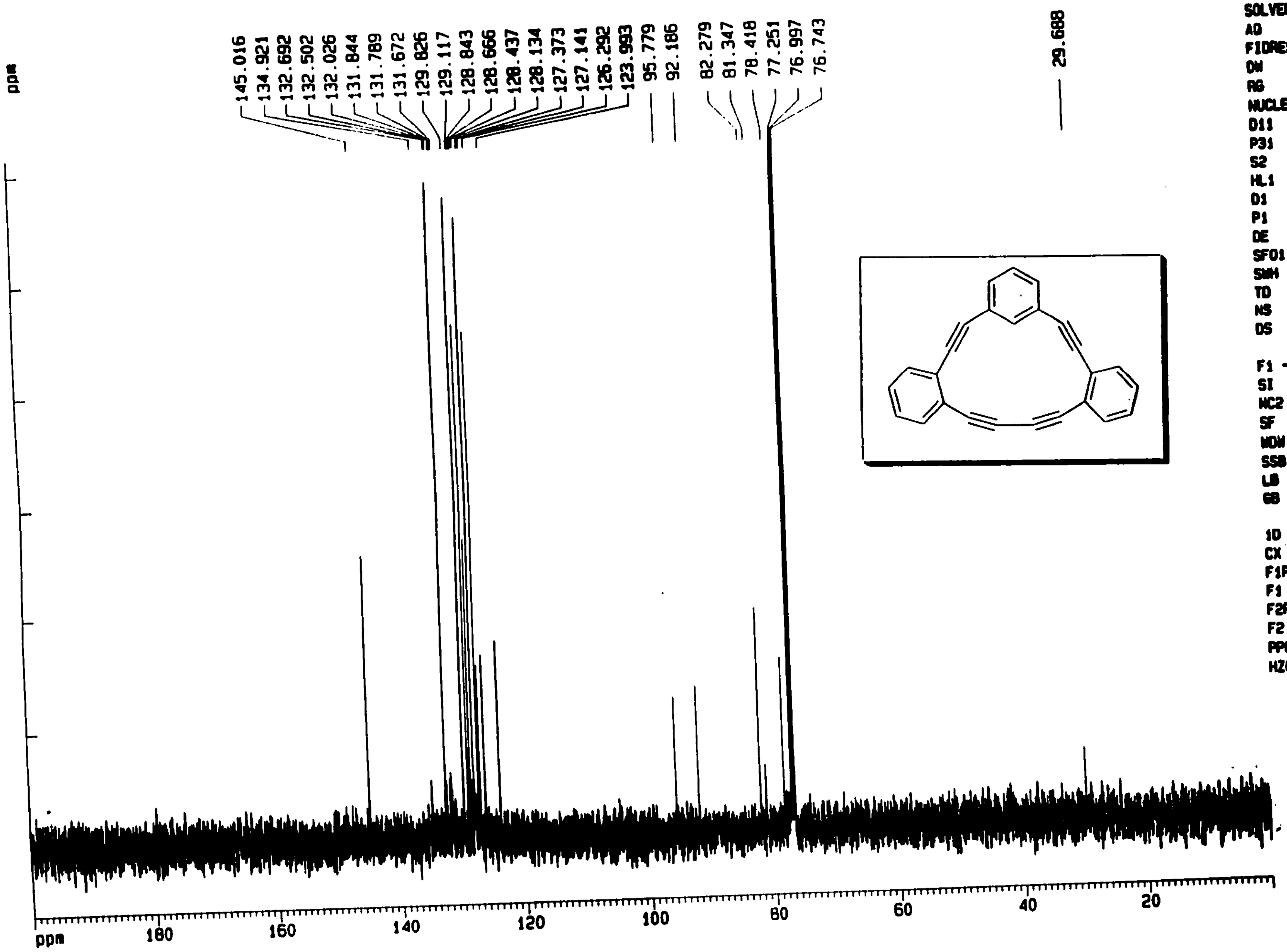
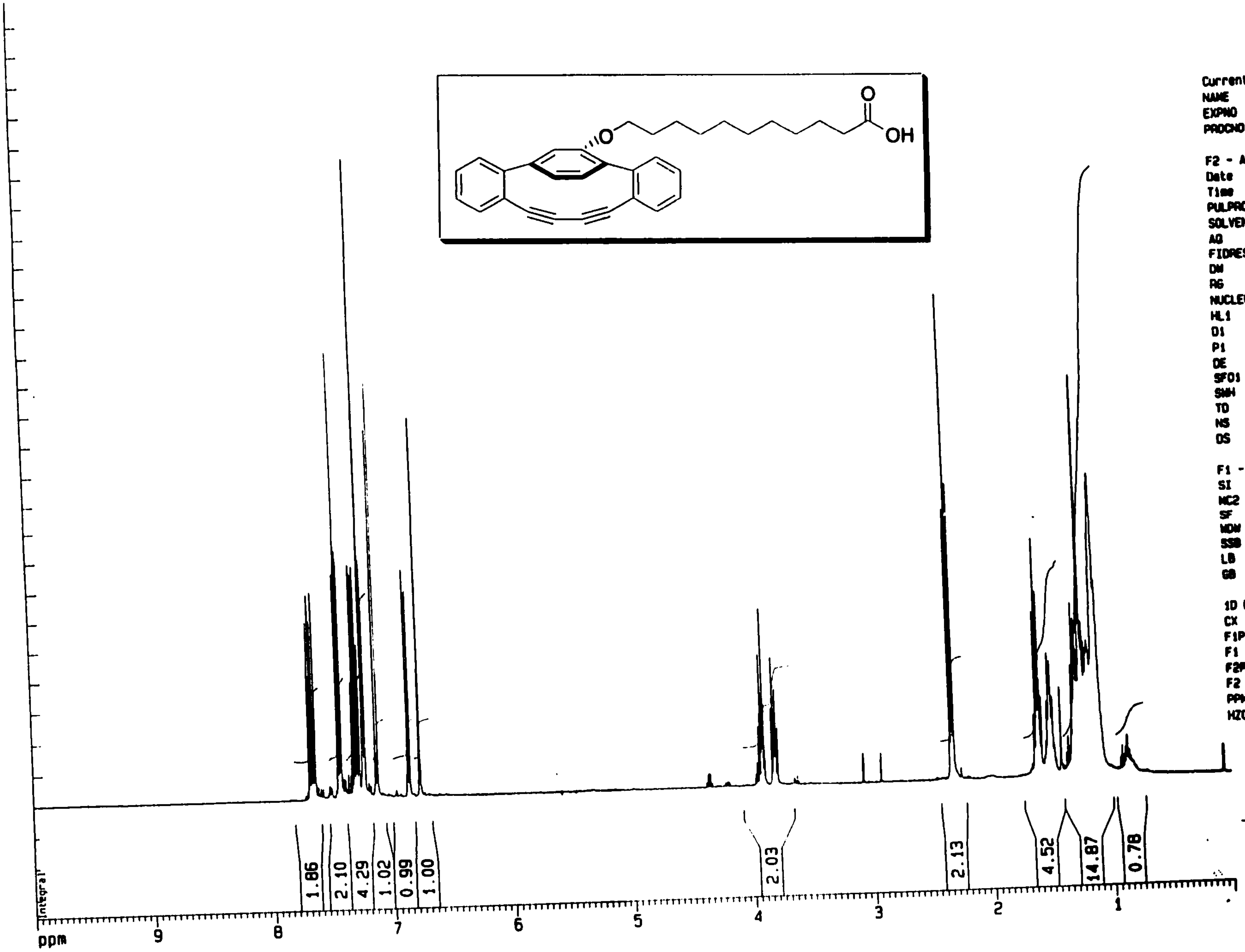


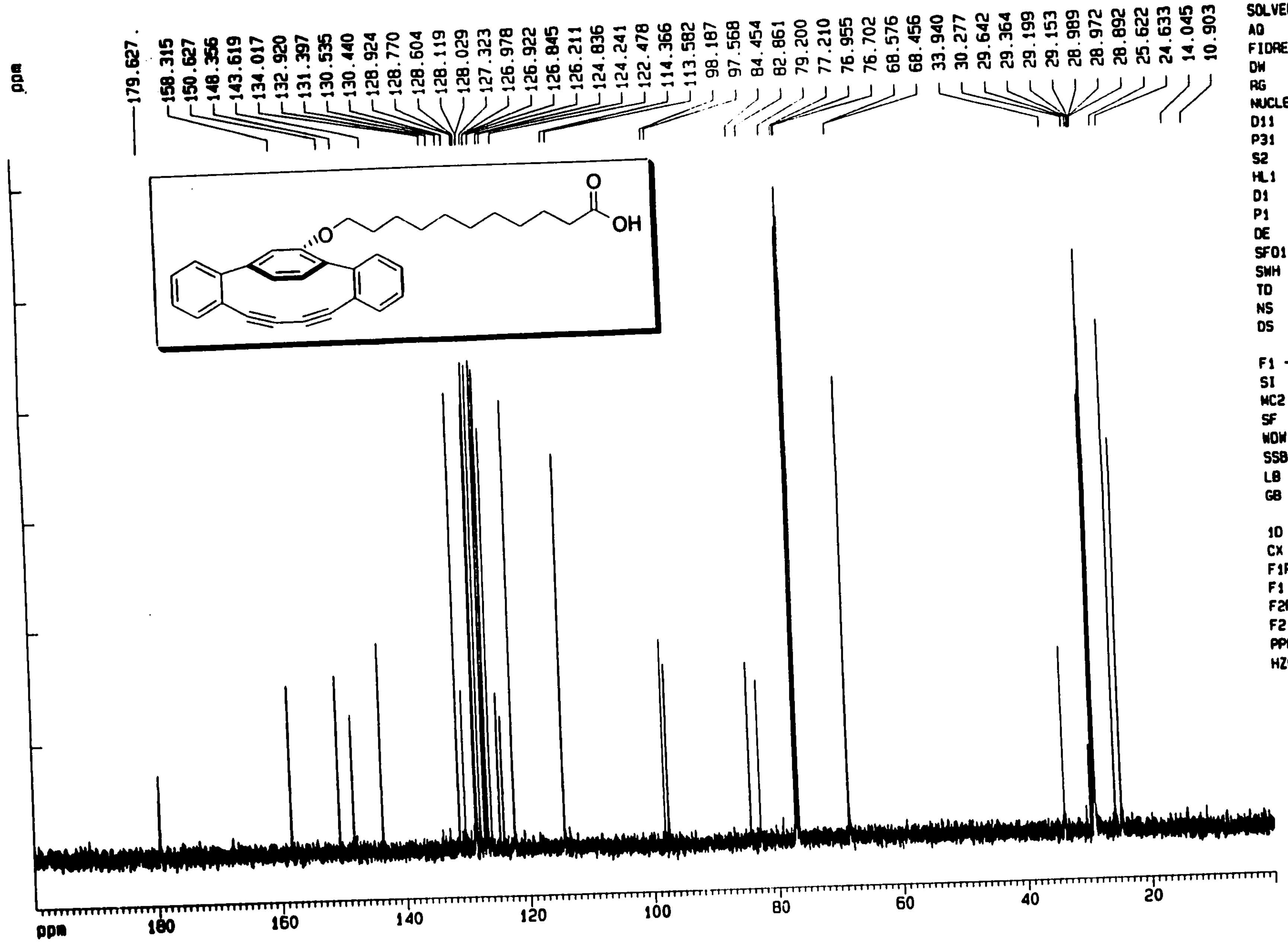
```
Current Data Parameters
NAME              shawn_991a
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date                20010308
Time                    4.46
PULPROG                   zg
SOLVENT                CDCl3
AQ              4.6530805 sec
FIDRES           0.107456 Hz
DW                  71.0 usec
RG                      1024
NUCLEUS                   1H
HL1                     0 dB
D1             0.0100000 sec
P1                   3.0 usec
DE                  88.8 usec
SFO1          500.1381707 MHz
SWH               7042.25 Hz
TD                     65536
NS                        16
DS                         0

F1 - Processing parameters
SI                     32768
MC2                       QF
SF            500.1354311 MHz
WDW                       EM
SSB                        0
LB                   0.00 Hz
GB                         0

1D NMR plot parameters
CX                  22.00 cm
F1P               10.000 ppm
F1                5001.35 Hz
F2P                0.000 ppm
F2                   0.00 Hz
PPMCM             0.45455 ppm/
HZCM            227.33429 Hz/(
```



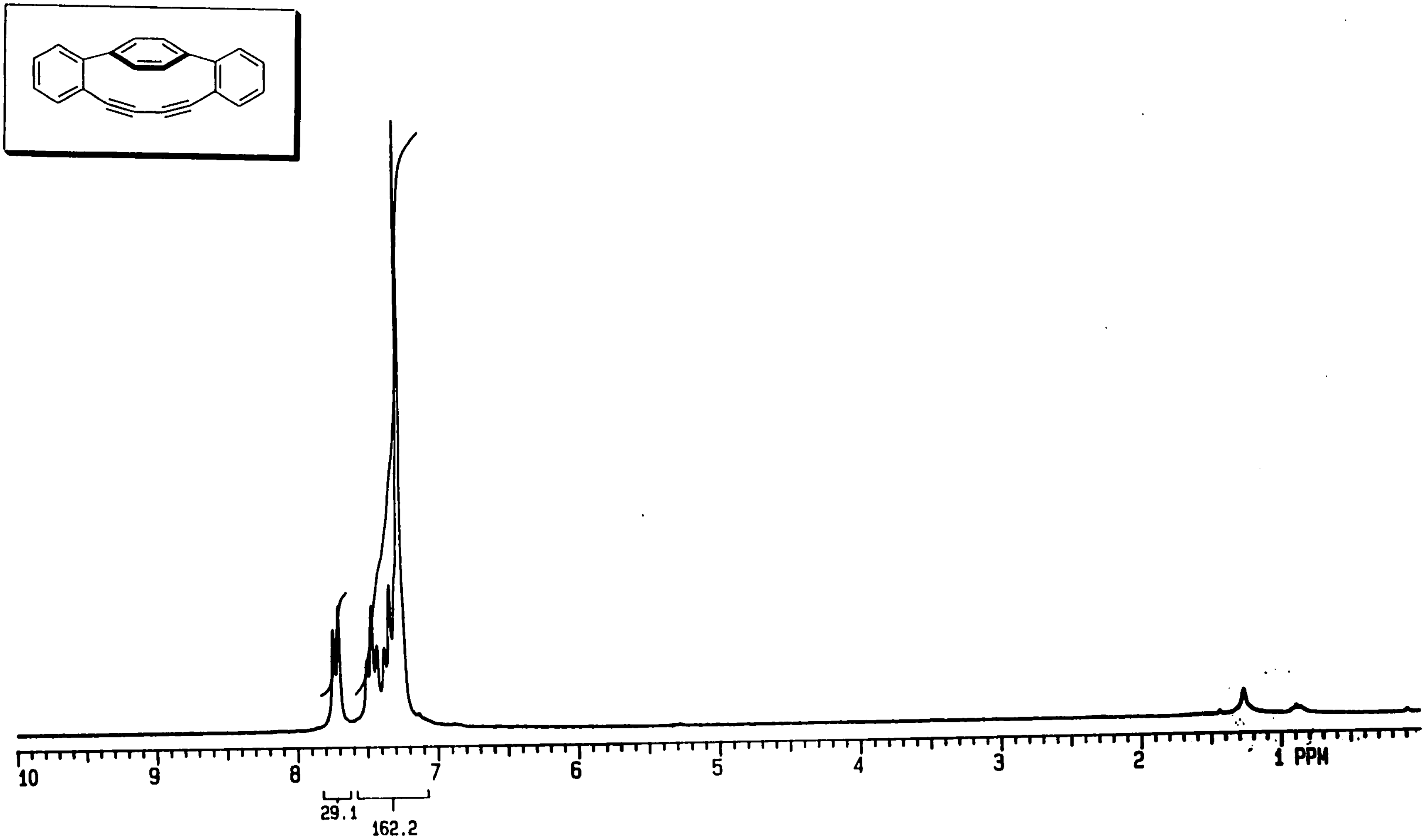
```
EXPNO                  2
PROCNO                 1

F2 - Acquisition Params
Date            20010308
Time                5.16
PULPROG             zgdc
SOLVENT            CDCl3
AQ             1.0485760
FIDRES          0.476837
DW                  16.0
RG                 32768
NUCLEUS              13C
D11            0.0300000
P31                 70.0
S2                    22
HL1                   22
D1             1.0000000
P1                   5.0
DE                  20.0
SFO1         125.7724464
SWH             31250.00
TD                 65536
NS                   872
DS                     0

F1 - Processing paramet
SI                 32768
MC2                   QF
SF           125.7591504
WDW                   EM
SSB                    0
LB                  1.00
GB                     0

1D NMR plot parameters
CX                 22.00
F1P              200.000
F1              25151.83
F2P                0.000
F2                  0.00
PPMCM            9.09091
HZCM          1143.26501
```

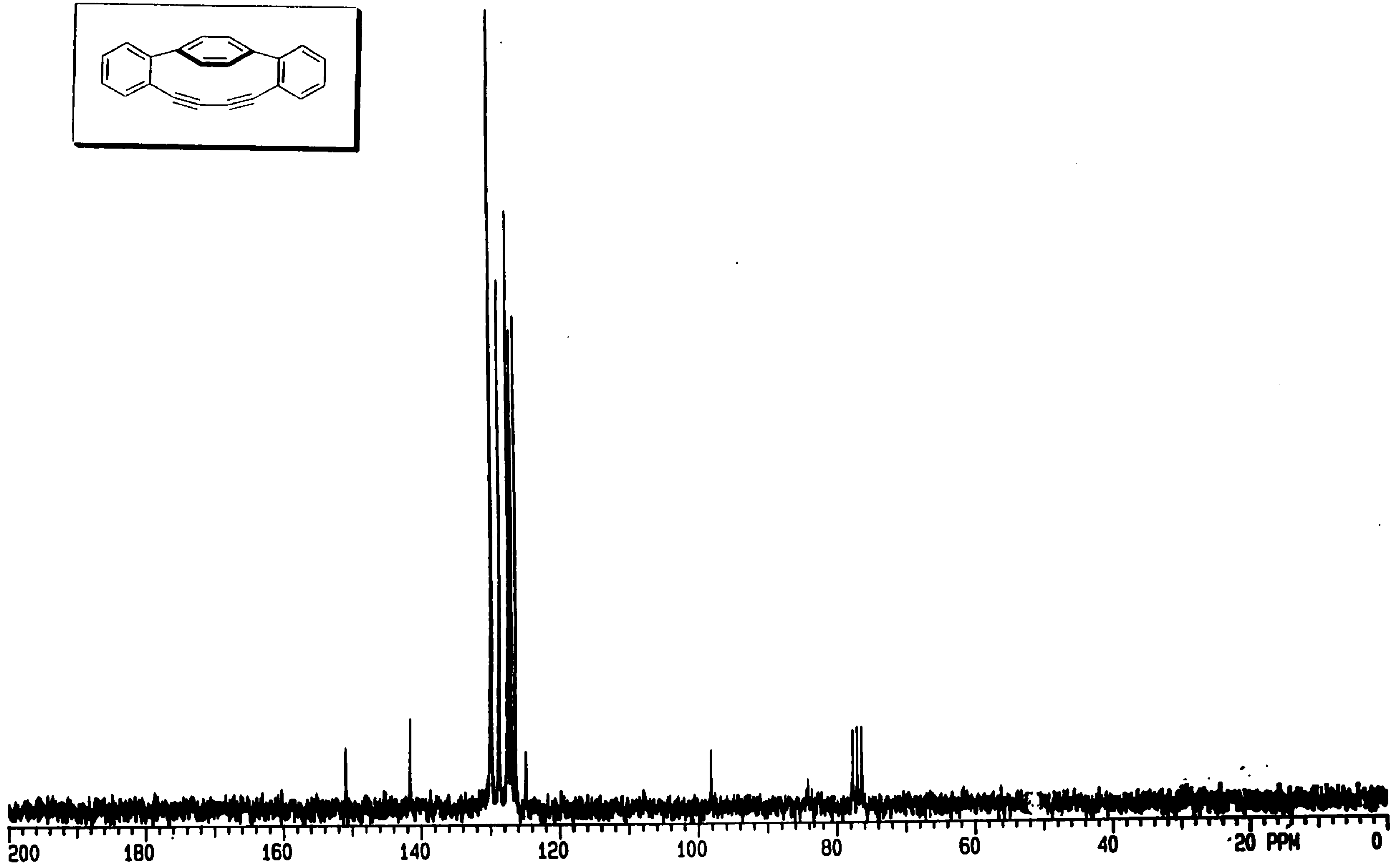


```
Current Data Parameters
NAME            shawn_551a
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date                991119
Time                 12.12
PULPROG                 zg
SOLVENT              CDCl3
AQ            4.6530805 sec
FIDRES         0.107456 Hz
DW                 71.0 use
RG                    1024
NUCLEUS                 1H
HL1                   0 dB
D1            0.0100000 sec
P1                  3.0 use
DE                 88.8 use
SFO1       500.1381707 MHz
SWH            7042.25 Hz
TD                   65536
NS                      16
DS                       0

F1 - Processing parameters
SI                   32768
MC2                     QF
SF         500.1354311 MHz
WDW                     EM
SSB                      0
LB                 0.00 Hz
GB                       0

1D NMR plot parameters
CX                22.00 cm
F1P             10.000 ppm
F1              5001.35 Hz
F2P              0.000 ppm
F2                 0.00 Hz
PPMCM          0.45455 ppm/
HZCM         227.33429 Hz/
```

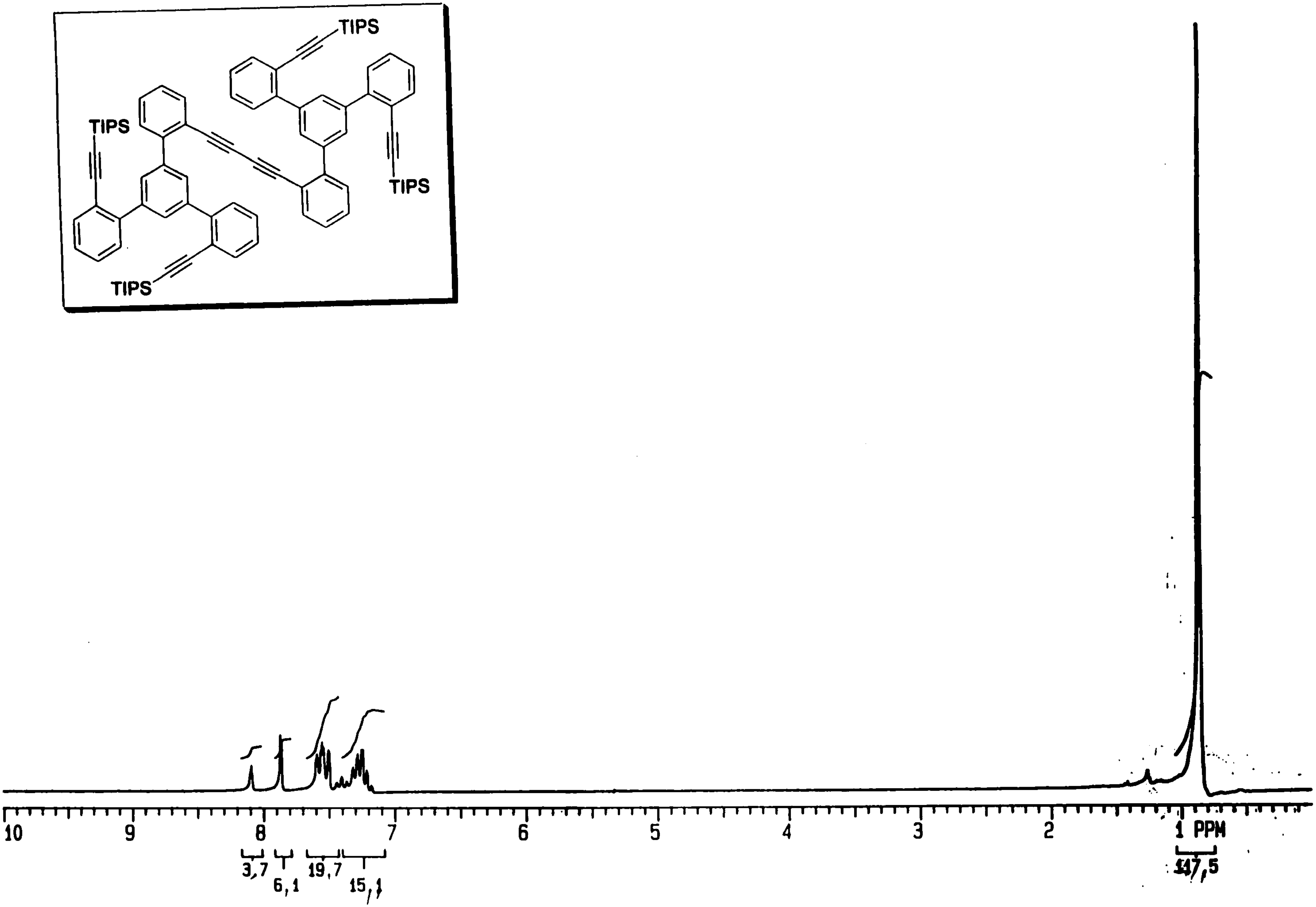


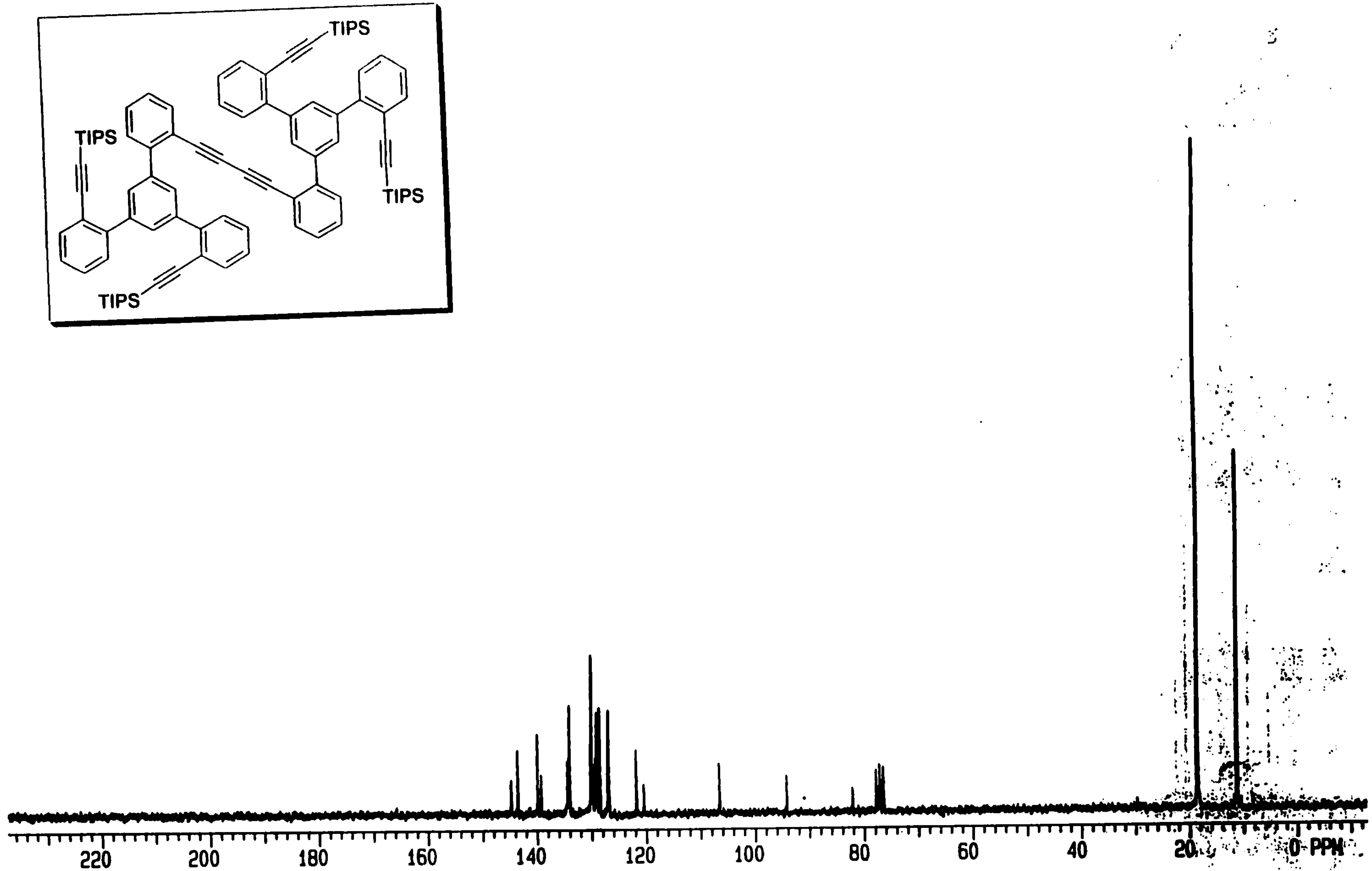
```
NAME            shawn_551a
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parame
Date                991119
Time                 12.46
PULPROG               zgdc
SOLVENT              CDC13
AQ               1.0485960
FIDRES            0.476837
DW                    16.0
RG                   32768
NUCLEUS                13C
D11              0.0300000
P31                   70.0
S2                      22
HL1                     22
D1               1.0000000
P1                     5.0
DE                    20.0
SFO1           125.7724464
SWH               31250.00
TD                   65536
NS                    1488
DS                       0

F1 - Processing paramet
SI                   32768
MC2                     QF
SF             125.7591571
WDW                     EM
SSB                      0
LB                    1.00
GB                       0

1D NMR plot parameters
CX                   22.00
F1P                200.000
F1                25151.83
F2P                  0.000
F2                    0.00
PPMCM              9.09091
HZCM            1143.26501
```

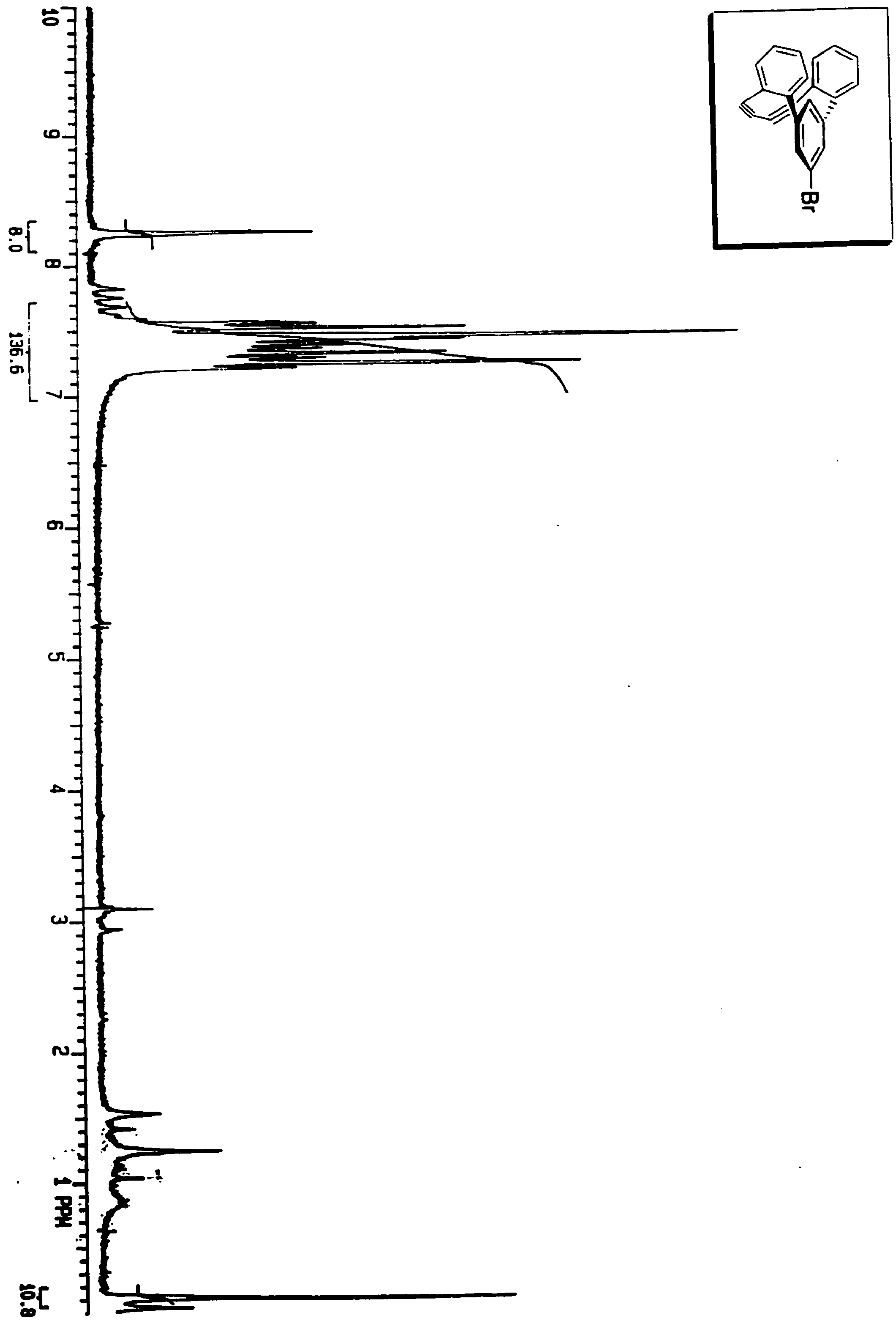

10
9
8
7
6
5
4
3
2
1 PPM
29.1
162.2

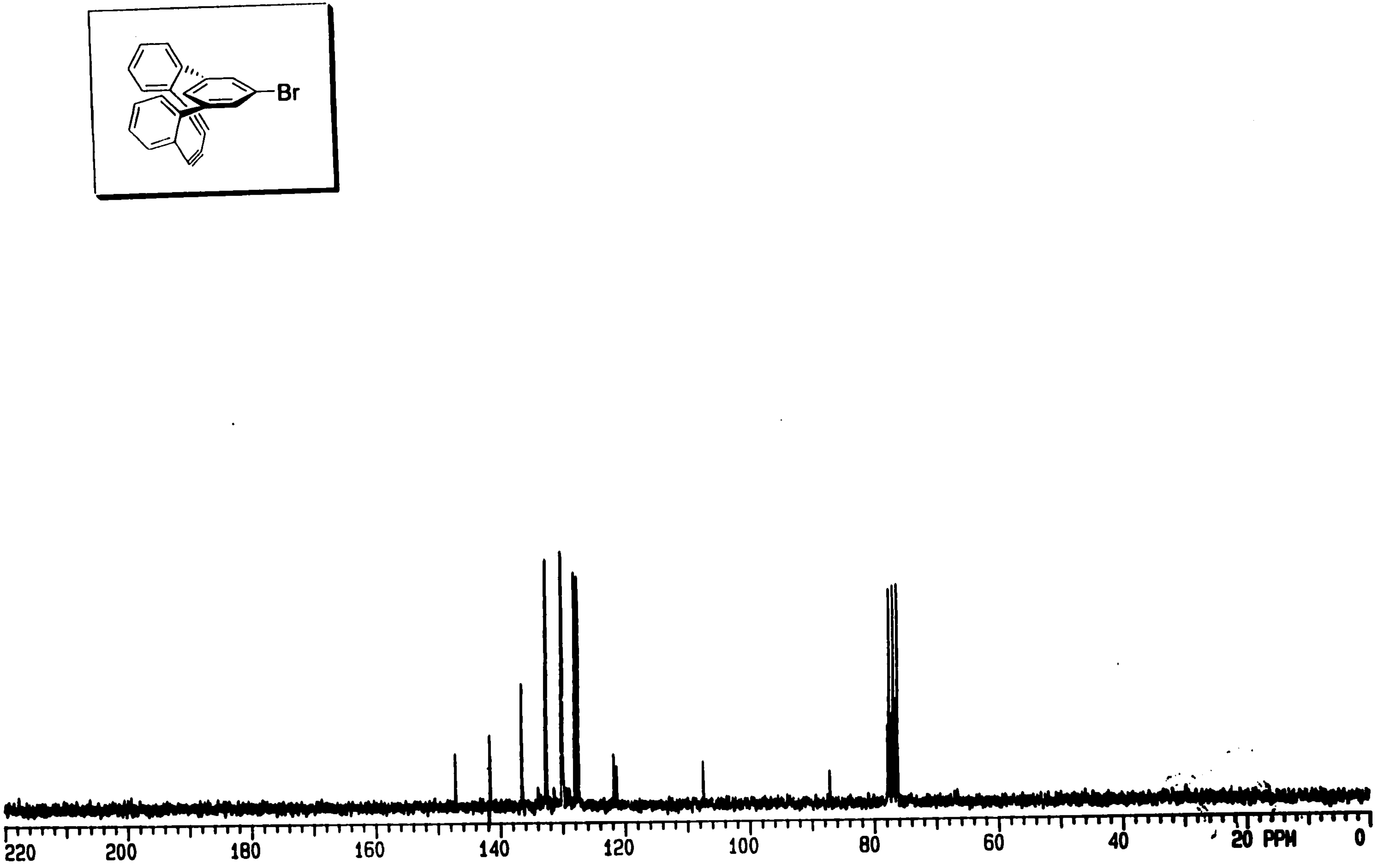


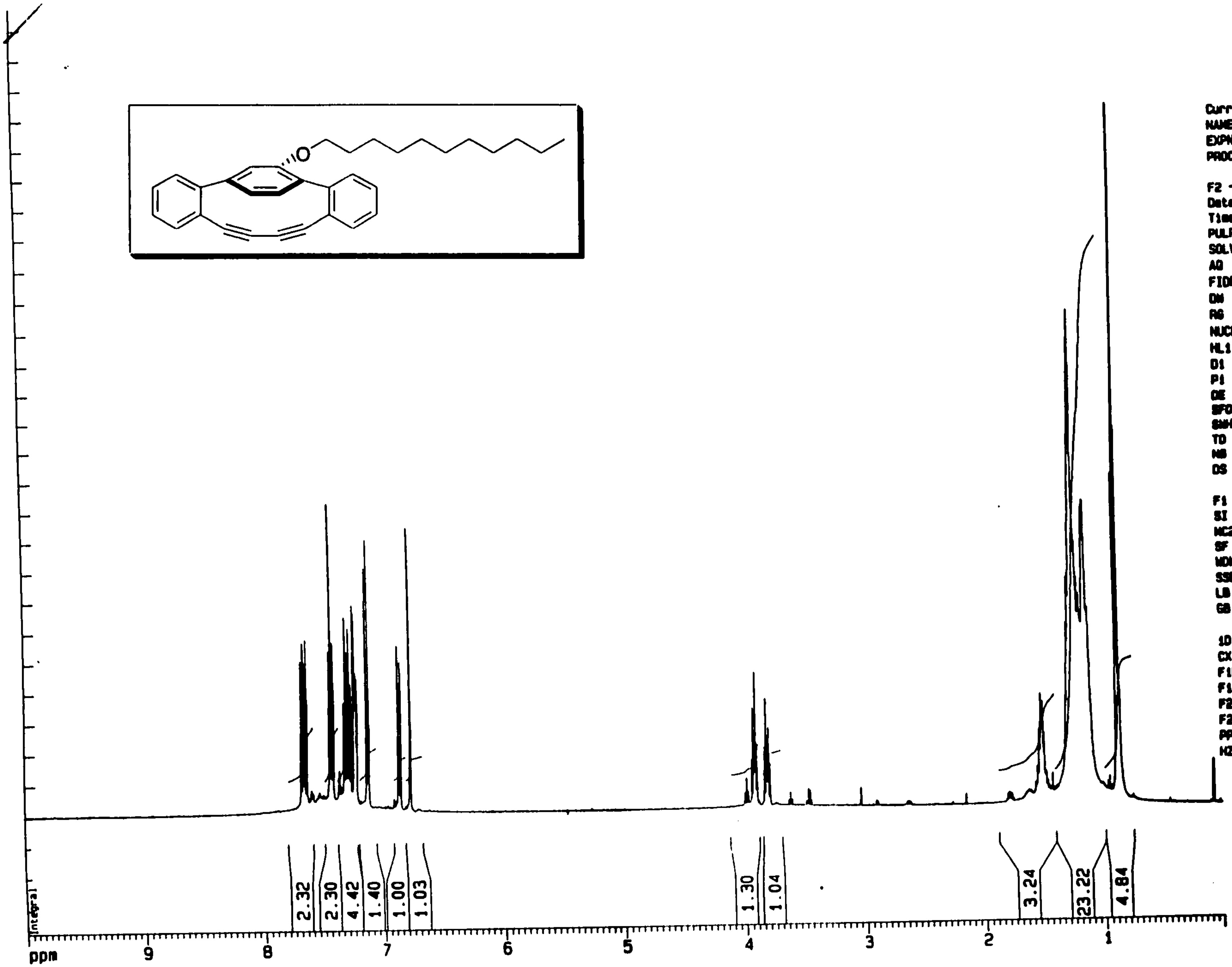
200
180
160
140
120
100
80
60
40
20 PPM
0


TIPS
TIPS
TIPS
TIPS
10
9
8
7
6
5
4
3
2
1 PPM
3,7
6,1
19,7
15,1
147,5

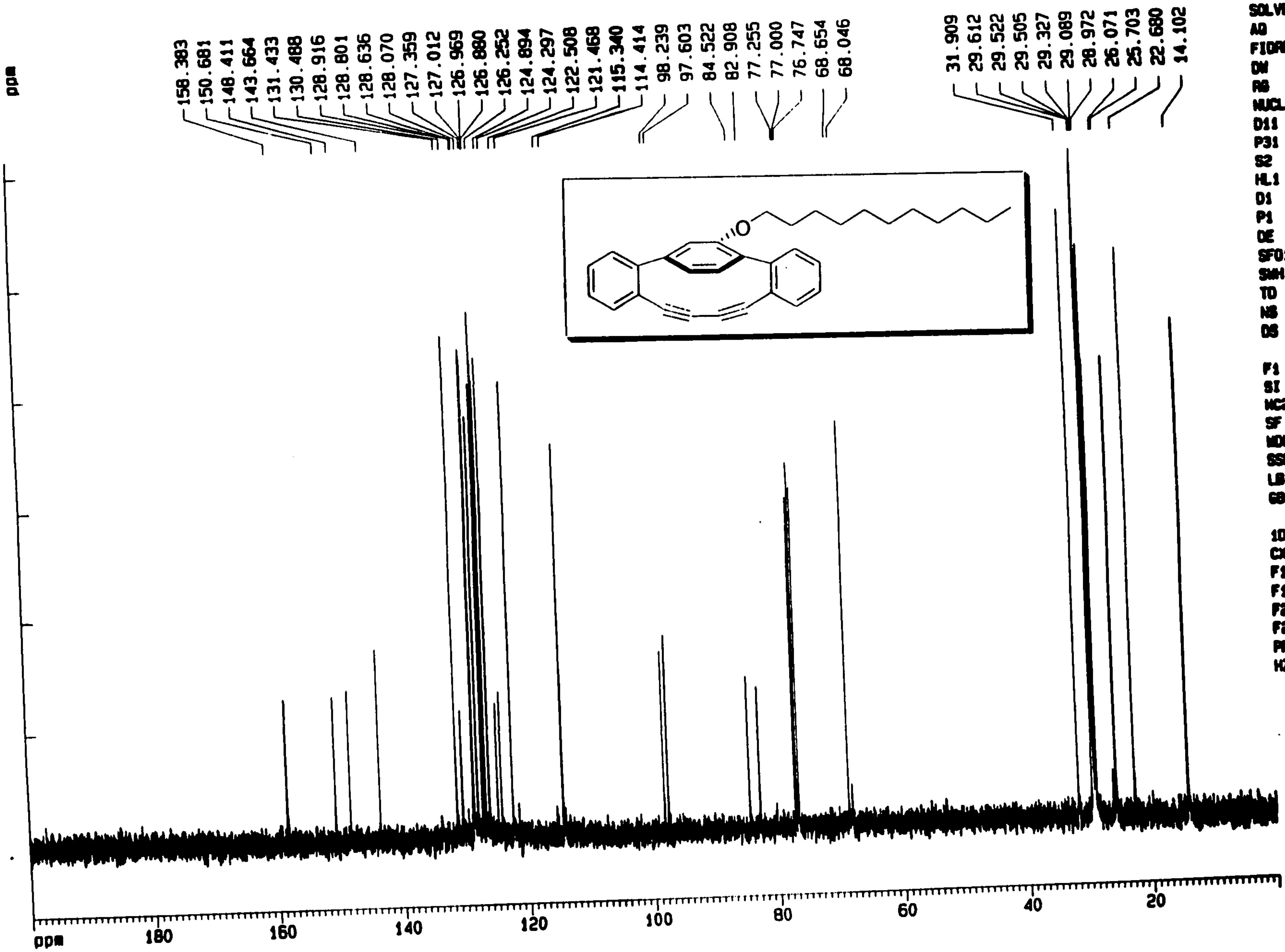

TIPS
TIPS
TIPS
TIPS
220
200
180
160
140
120
100
80
60
40
20
0 PPM


10
9
8
7
6
5
4
3
2
1 PPM
8.0
136.6
10.8
Br

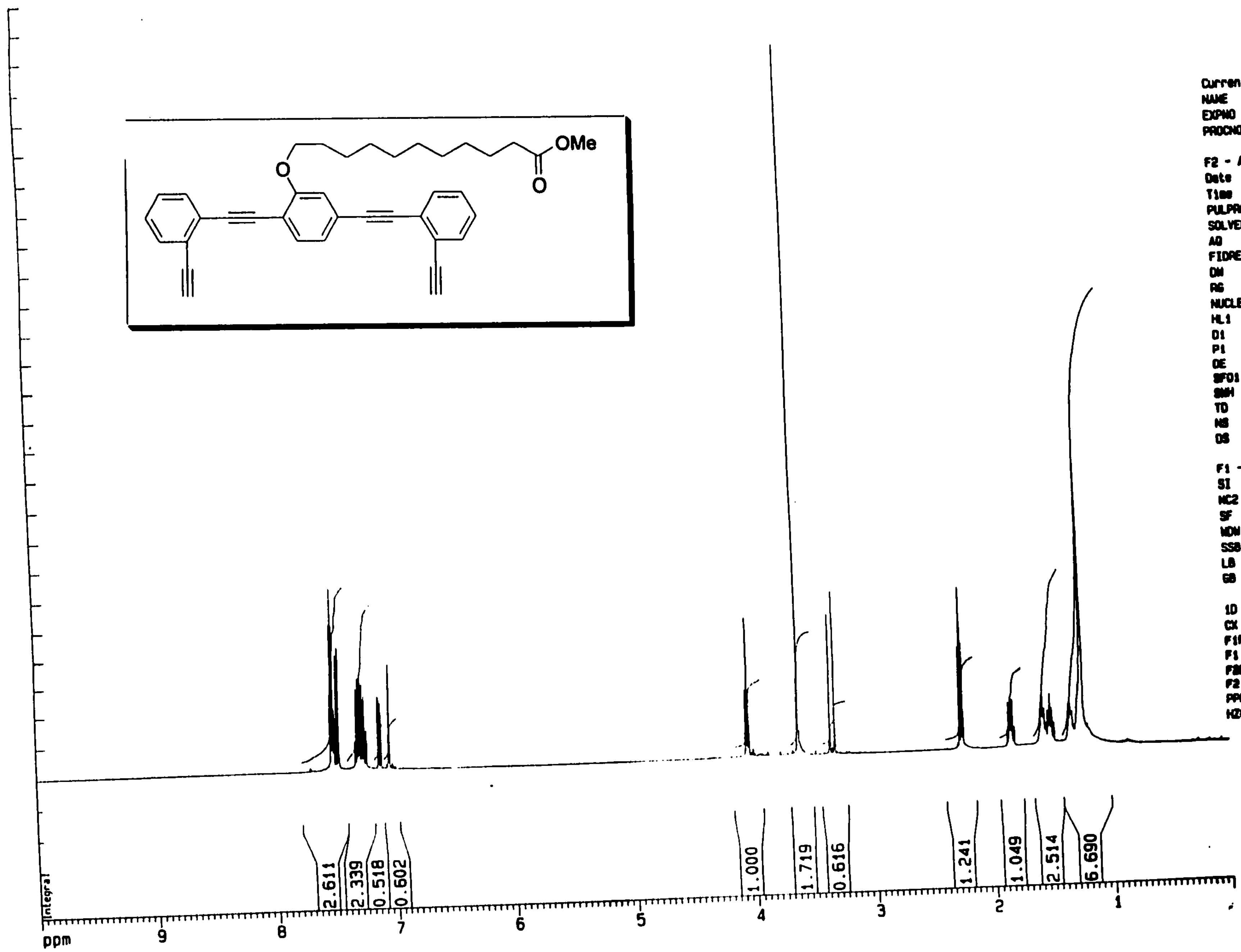

Br
220
200
180
160
140
120
100
80
60
40
20 PPM
0



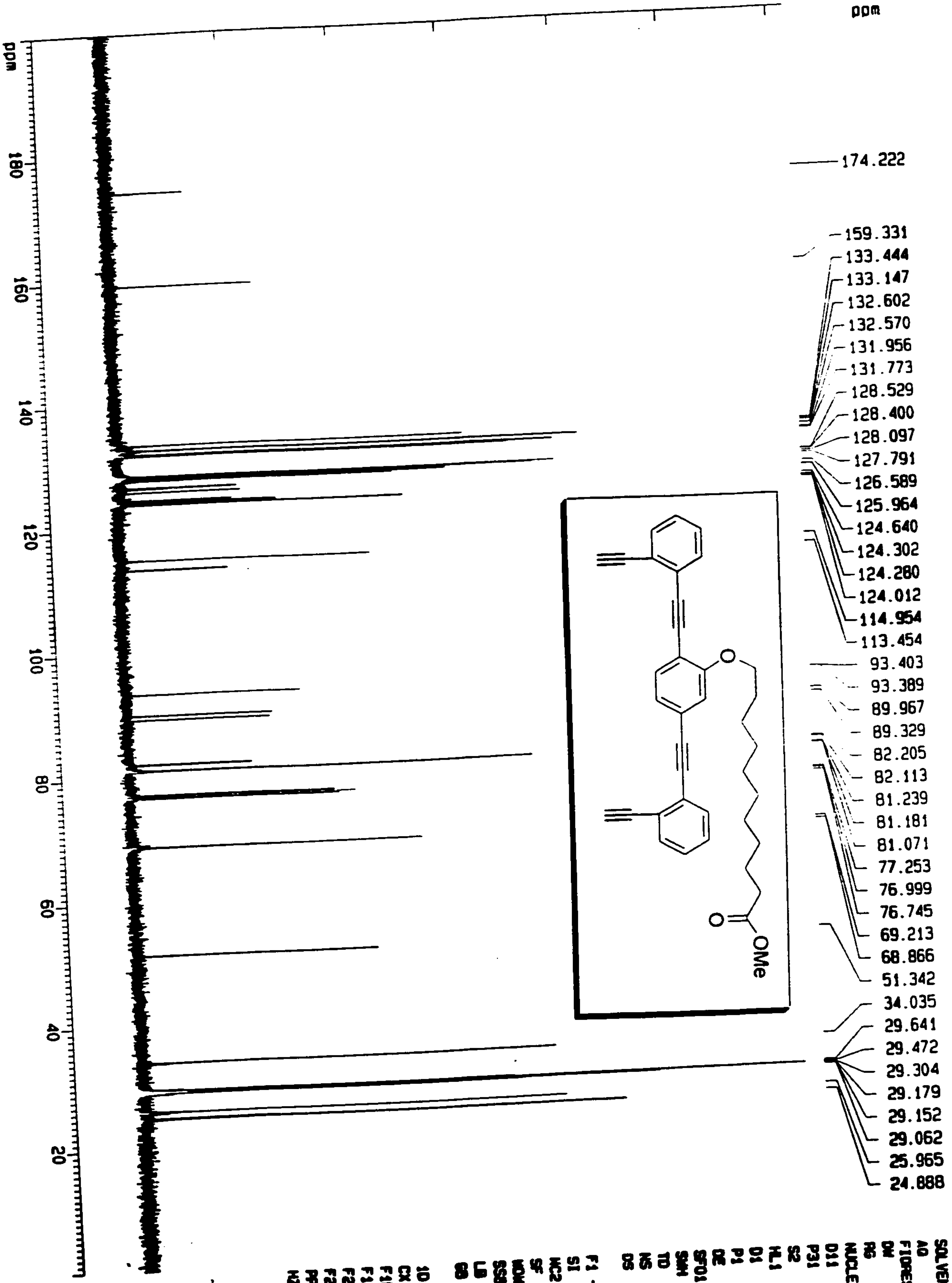
```
Current Data Parameters
NAME             shawn_549a
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date                 981129
Time                  15.23
PULPROG                  zg
SOLVENT               CDCl3
AQ             4.6530805 sec
FIDRES          0.107456 Hz
DW                  71.0 usec
RG                      512
NUCLEUS                  1H
NL1                    0 dB
D1             0.0100000 sec
P1                   3.0 usec
DE           [illegible] usec
SFO1       500.1381707 MHz
SWH              7042.25 Hz
TD                    65536
NS                       16
DS                        0

F1 - Processing parameters
SI                    32768
MC2                      QF
SF          500.1354311 MHz
WDW                      EM
SSB                       0
LB                  0.00 Hz
GB                        0

1D NMR plot parameters
CX                  22.00 cm
F1P               10.000 ppm
F1               5001.35 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.45455 ppm/
HZCM           227.33429 Hz/(
```



```
NAME          shawn_549a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parame
Date              991129
Time               15.41
PULPROG             zgdc
SOLVENT            CDCl3
AQ             1.0485960
FIDRES          0.476837
DW                  16.0
RG                 32768
NUCLEUS              13C
D11            0.0300000
P31                 70.0
S2                    22
HL1                   22
D1             1.0000000
P1                   5.0
DE                  20.0
SFO1         125.7724464
SWH             31250.00
TD                 65536
NS                   512
DS                     0

F1 - Processing paramet
SI                 32768
MC2                   QF
SF           125.7591523
WDW                   EM
SSB                    0
LB                  1.00
GB                     0

1D NMR plot parameters
CX                 22.00
F1P              200.000
F1              25151.83
F2P                0.000
F2                  0.00
PPMCM            9.09091
HZCM          1143.28501
```



```
Current Data Parameters
NAME            shawn_555a
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date                991123
Time                 15.30
PULPROG                 zg
SOLVENT              CDC13
AQ            4.6530805 sec
FIDRES         0.107456 Hz
DW                71.0 usec
RG                     512
NUCLEUS                 1H
HL1                   0 dB
D1            0.0100000 sec
P1                 3.0 usec
DE         [illegible] usec
SFO1       500.1381707 MHz
SWH             7042.25 Hz
TD                   65536
NS                      16
DS                       0

F1 - Processing parameters
SI                   32768
MC2                     QF
SF         500.1354311 MHz
WDW                     EM
SSB                      0
LB                 0.00 Hz
GB                       0

1D NMR plot parameters
CX                22.00 cm
F1P             10.000 ppm
F1              5001.36 Hz
F2P              0.000 ppm
F2                 0.00 Hz
PPMCM          0.45455 ppm/
HZCM         227.33429 Hz/(
```



EXPNO	2
PROCNO	1

F2 - Acquisition Parame

Date	991123
Time	15.47
PULPROG	zgdc
SOLVENT	CDCl3
AQ	1.0485950
FIDRES	0.476837
DW	16.0
RG	32768
NUCLEUS	13C
D11	0.030000
P31	70.0
S2	22
HL1	22
D1	1.000000
P1	5.0
DE	20.0
SFO1	125.772464
SWH	31250.00
TD	65536
NS	512
DS	0

F1 - Processing paramet

SI	32768
MC2	QF
SF	125.7591571
WDW	EM
SSB	0
LB	1.00
GB	0

1D NMR plot parameters

CX	22.00
F1P	200.000
F1	25151.83
F2P	0.000
F2	0.00
PPMCM	9.09091
HZCM	1143.26501

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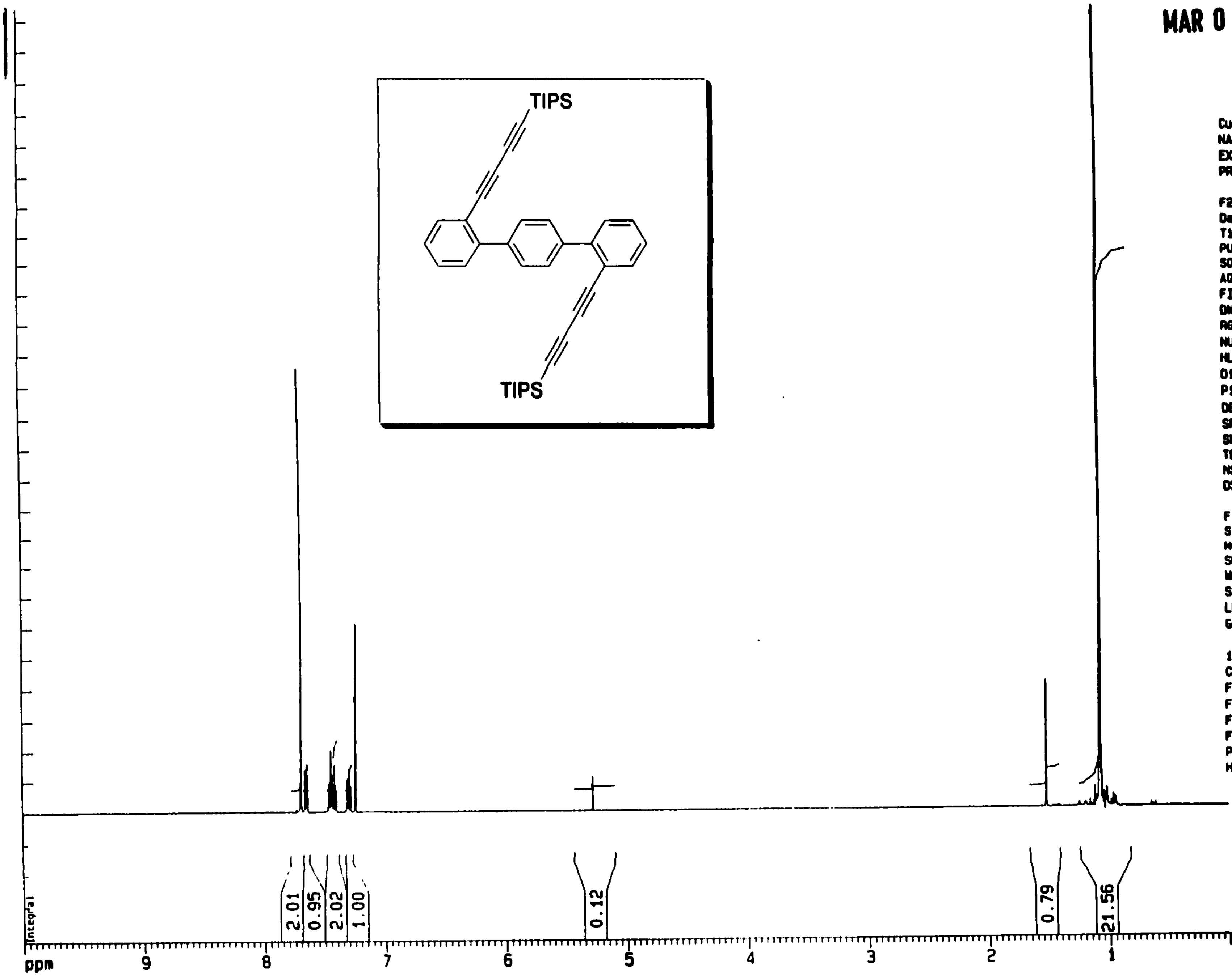
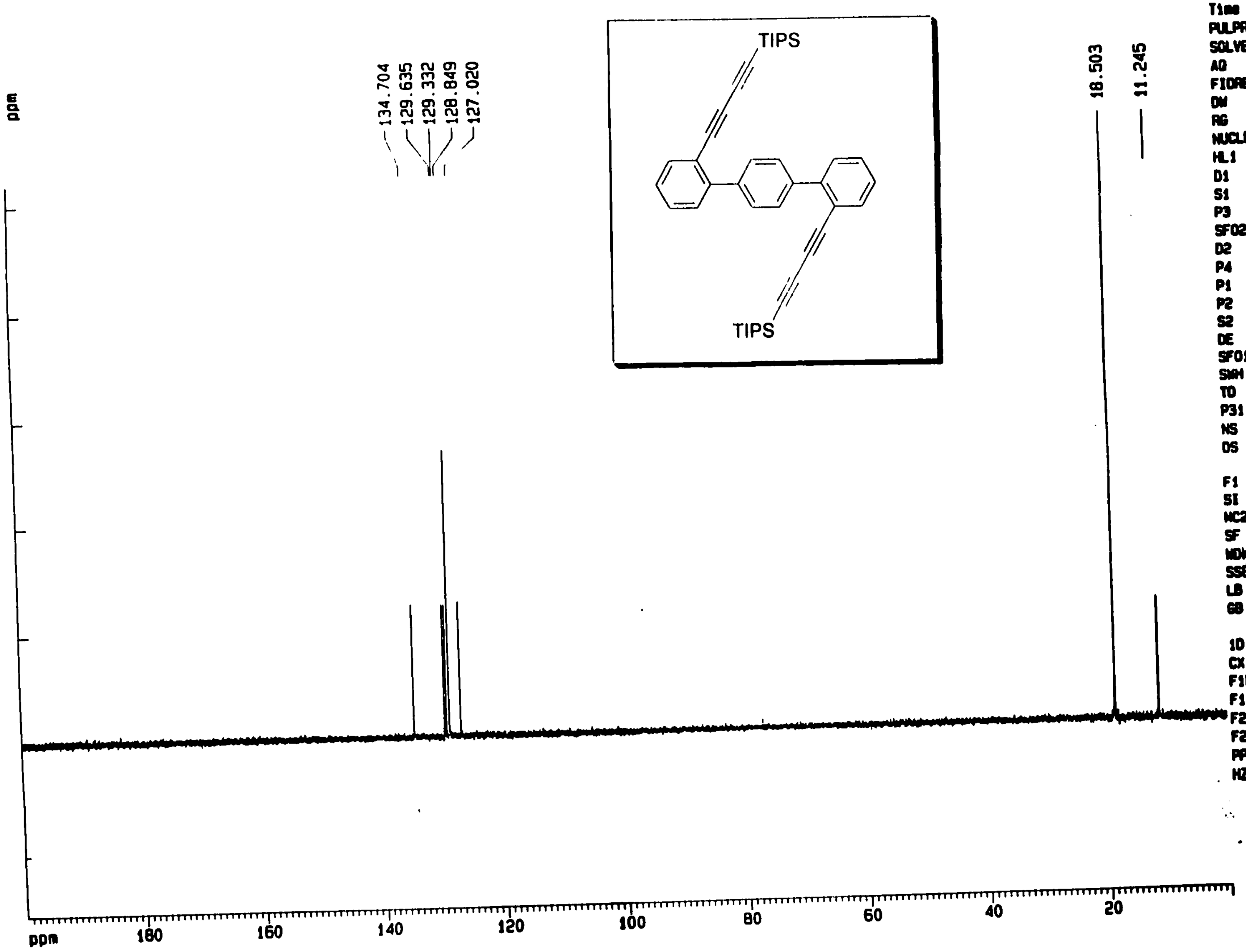


Current Data Parameters	
NAME	shawn_981
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date	20010224
Time	12.55
PULPROG	zg
SOLVENT	CDCl3
AQ	4.6530805 sec
FIDRES	0.107456 Hz
DW	71.0 usec
RG	512
NUCLEUS	1H
HL1	0 dB
D1	0.0100000 sec
P1	3.0 usec
DE	88.8 usec
SFO1	500.1381707 MHz
SWH	7042.25 Hz
TD	65536
NS	16
DS	0
F1 - Processing parameters	
SI	32768
MC2	QF
SF	500.1354311 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
1D NMR plot parameters	
CX	22.00 cm
F1P	10.000 ppm
F1	5001.36 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.45455 ppm/
HZCM	227.33429 Hz/c

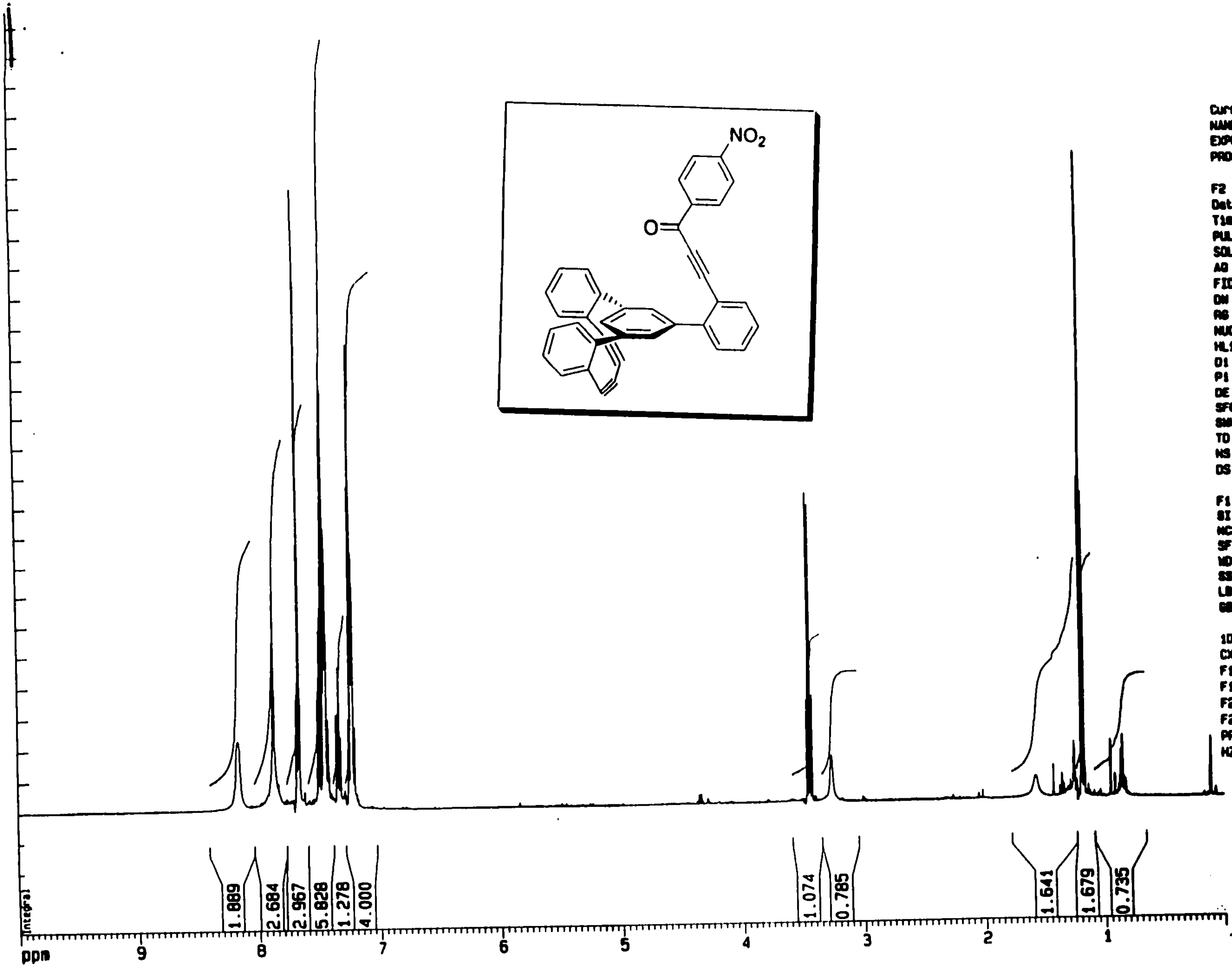


```
NAME
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parame
Date            20010226
Time                5.43
PULPROG         dept135
SOLVENT            CDCl3
AQ             1.0485960
FIDRES          0.476837
DW                  16.0
RG                 32768
NUCLEUS              13C
HL1                   22
D1             1.0000000
S1                     0
P3                   5.3
SFO2         500.1381707
D2             0.0035000
P4                  10.6
P1                   9.9
P2                  19.8
S2                    22
DE                  20.0
SFO1         125.7724464
SWH             31250.00
TD                 65536
P31                 70.0
NS                 10791
DS                     0

F1 - Processing paramet
SI                 32768
MC2                   QF
SF           125.7591571
WDW                   EM
SSB                    0
LB                  1.00
GB                     0

1D NMR plot parameters
CX                 22.00
F1P              135.712
F1              17067.09
F2P              125.545
F2              15788.40
PPMCM            0.46217
HZCM            58.12237
```

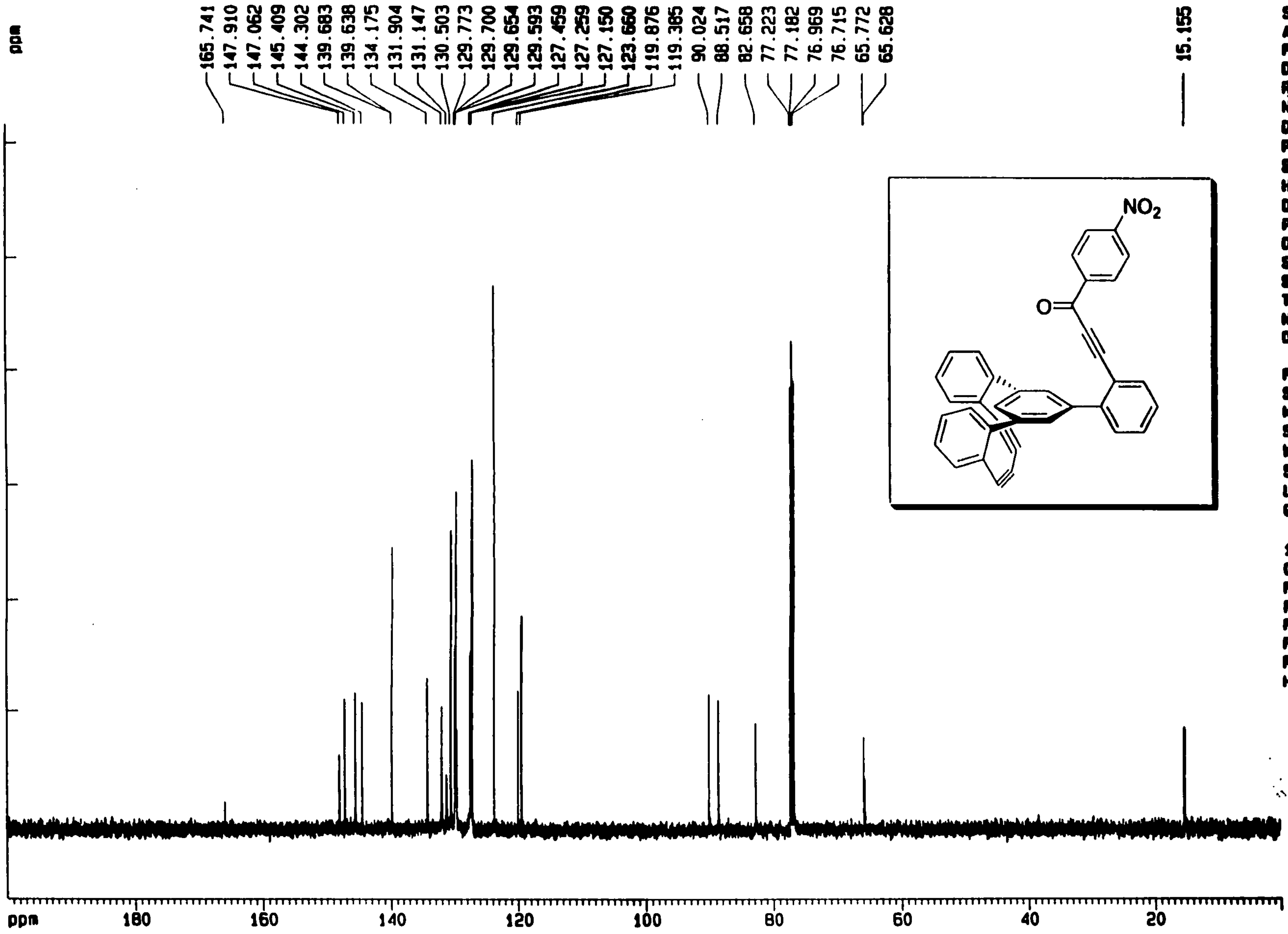


```
Current Data Parameters
NAME           shawn_B05a
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date             20000912
Time                 6.04
PULPROG                zg
SOLVENT             CDCl3
AQ           4.6530806 sec
FIDRES        0.107456 Hz
DW               71.0 usec
RG                   2048
NUCLEUS                1H
HL1                  0 dB
D1           0.0100000 sec
P1                3.0 usec
DE               88.8 usec
SFO1      500.1381707 MHz
SWH            7042.25 Hz
TD                  65536
NS                     16
DS                      0

F1 - Processing parameters
SI                  32768
MC2                    QF
SF        500.1354311 MHz
WDW                    EM
SSB                     0
LB                0.00 Hz
GB                      0

1D NMR plot parameters
CX               22.00 cm
F1P             8.568 ppm
F1             4285.17 Hz
F2P             6.897 ppm
F2             3449.42 Hz
PPMCM         0.07594 ppm/
HZCM         37.97947 Hz/(
```



Current Data Parameters	
NAME	shawn_B05a
EXPNO	2
PROCNO	1

F2 - Acquisition Parame	
Date	20000912
Time	6.36
PULPROG	zgdc
SOLVENT	CDCl3
AQ	1.0485760
FIDRES	0.476837
DW	16.0
RG	32768
NUCLEUS	13C
D11	0.0300000
P31	70.0
S2	22
HL1	22
D1	1.0000000
P1	5.0
DE	20.0
SFO1	125.7724464
SWH	31250.00
TD	65536
NS	970
DS	0

F1 - Processing paramet	
SI	32768
MC2	QF
SF	125.7591571
WDW	EM
SSB	0
LB	1.00
GB	0

1D NMR plot parameters	
CX	22.00
F1P	200.000
F1	25151.83
F2P	0.000
F2	0.00
PPMCM	9.09091
HZCM	1143.26501

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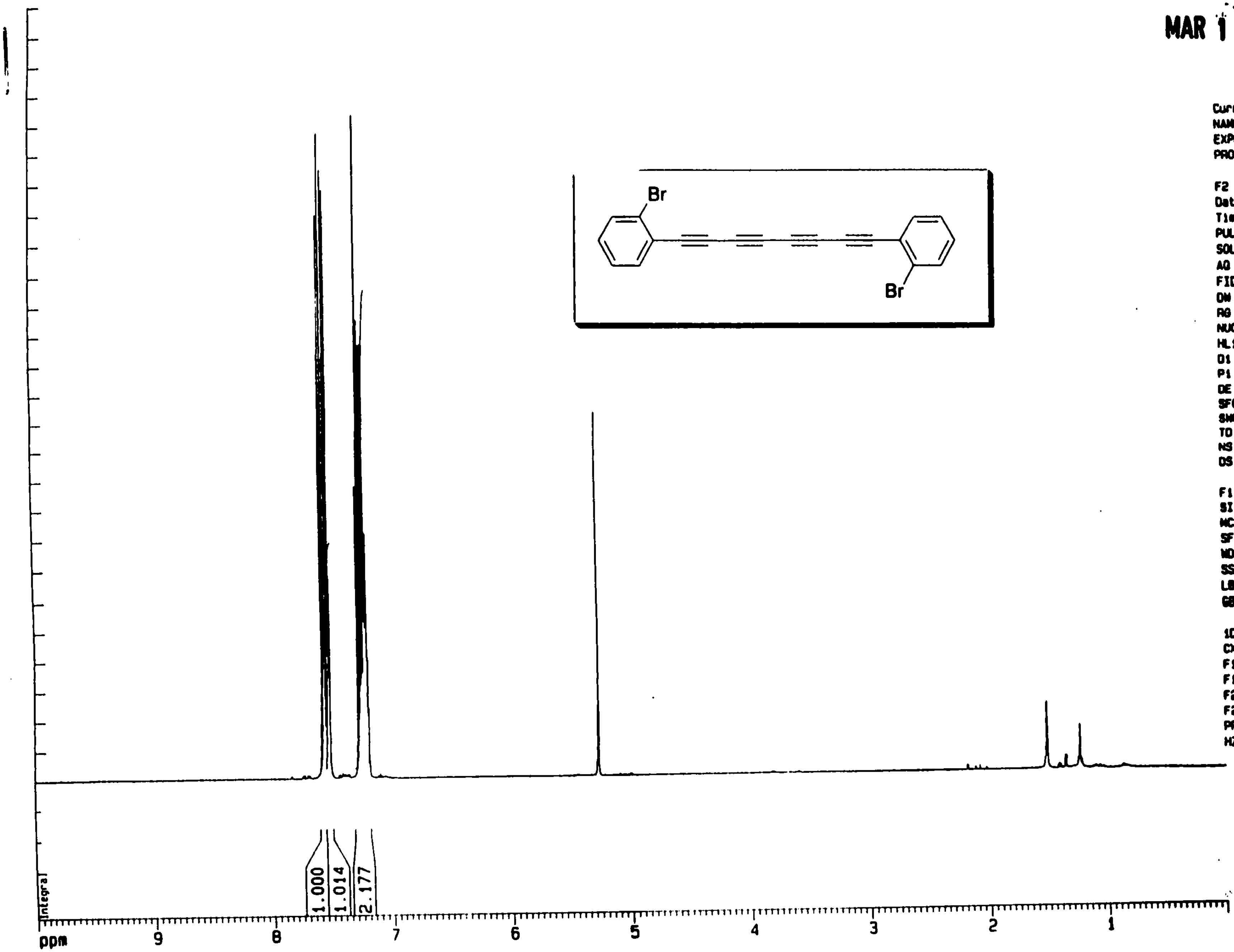
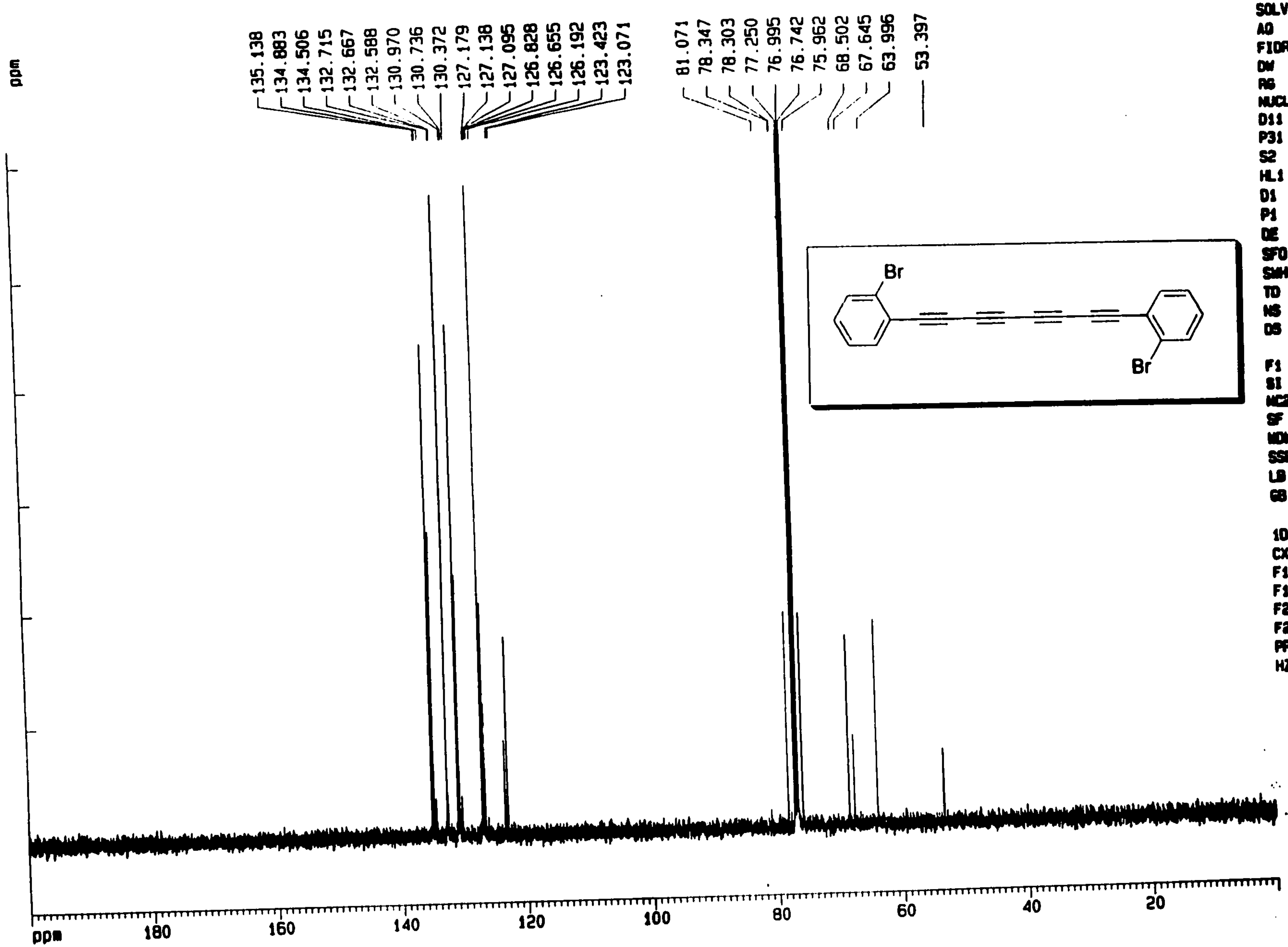


```
Current Data Parameters
NAME          shawn_993a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date            20010308
Time               13.59
PULPROG               zg
SOLVENT            CDCl3
AQ          4.6530805 sec
FIDRES       0.107456 Hz
DW             71.0 usec
RG                   512
NUCLEUS               1H
HL1                 0 dB
D1         0.0100000 sec
P1              3.0 usec
DE             80.0 usec
SFO1      500.1381707 MHz
SWH           7042.25 Hz
TD                 65536
NS                    16
DS                     0

F1 - Processing parameters
SI                 32768
MC2                   QF
SF        500.1354311 MHz
WDW                   EM
SSB                    0
LB               0.00 Hz
GB                     0

1D NMR plot parameters
CX              22.00 cm
F1P           10.000 ppm
F1            5001.36 Hz
F2P            0.000 ppm
F2               0.00 Hz
PPMCM         0.45455 ppm/
HZCM        227.33429 Hz/
```



Current Data Parameters

NAME	shawn_993a
EXPNO	3
PROCNO	1

F2 - Acquisition Parame

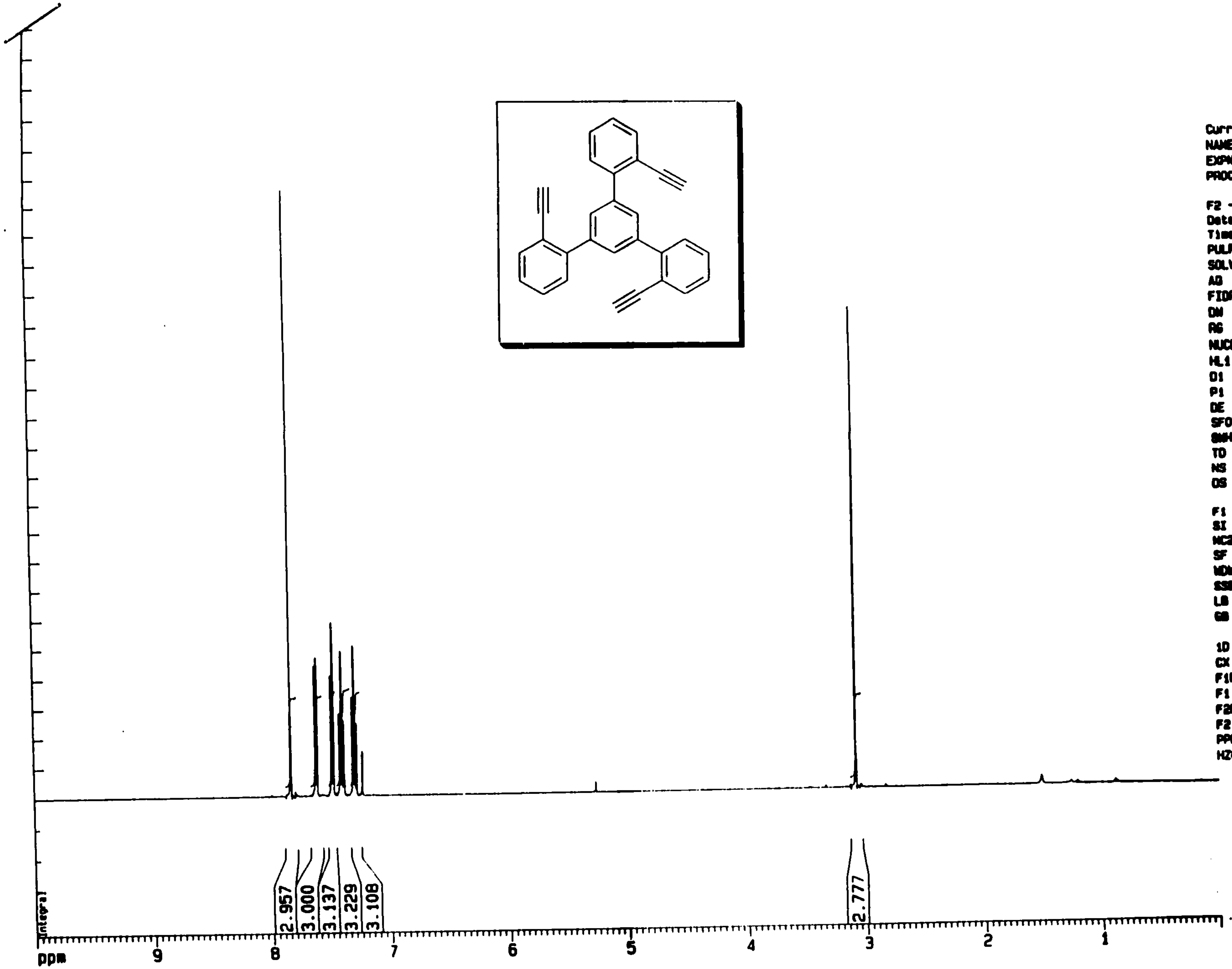
Date	20010308
Time	14.03
PULPROG	zgdc
SOLVENT	CDCl3
AQ	1.0485960
FIDRES	0.476837
DW	16.0
RG	32768
NUCLEUS	13C
D11	0.0300000
P31	70.0
S2	22
HL1	22
D1	5.0000000
P1	5.0
DE	20.0
SFO1	125.7724464
SWH	31250.00
TD	65536
NS	468
DS	0

F1 - Processing paramet

SI	32768
MC2	QF
SF	125.7591533
WDW	EM
SSB	0
LB	1.00
GB	0

1D NMR plot parameters

CX	22.00
F1P	200.000
F1	25151.83
F2P	0.000
F2	0.00
PPMCM	9.09091
HZCM	1143.26501

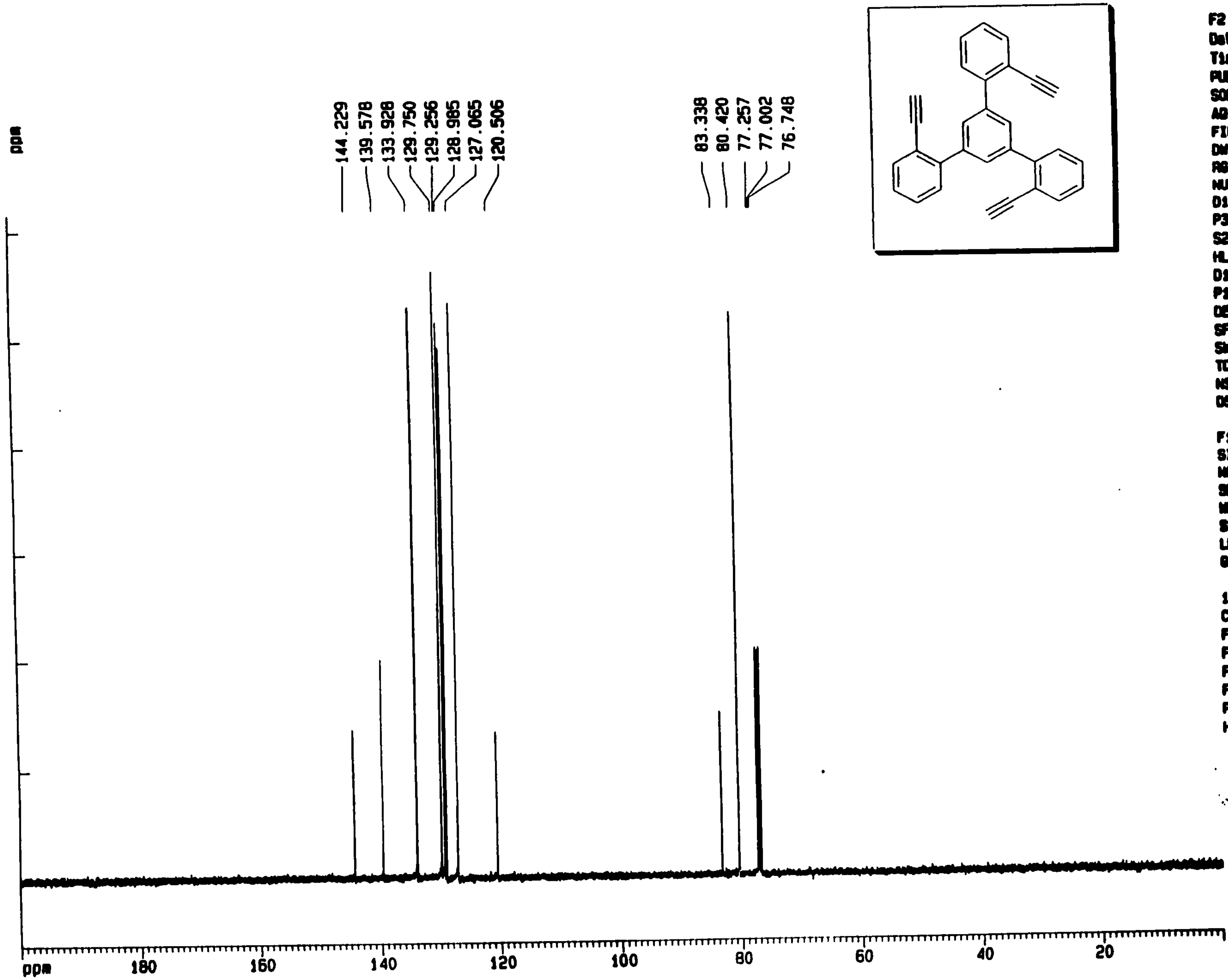


```
Current Data Parameters
NAME             shawn_311a
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date                 990301
Time                  12.46
PULPROG                  zg
SOLVENT               CDCl3
AQ             4.6530805 sec
FIDRES          0.107456 Hz
DW                  71.0 usec
RG                      256
NUCLEUS                  1H
HL1                       0 dB
D1             0.0100000 sec
P1                   3.0 usec
DE                  88.8 usec
SFO1         500.1381707 MHz
SWH              7042.25 Hz
TD                    65536
NS                       16
DS                        0

F1 - Processing parameters
SI                    32768
MC2                      QF
SF           500.1354311 MHz
WDW                      EM
SSB                       0
LB                     0.00 Hz
GB                        0

1D NMR plot parameters
CX                    22.00 cm
F1P                  10.000 ppm
F1                  5001.35 Hz
F2P                   0.000 ppm
F2                     0.00 Hz
PPMCM               0.45455 ppm/
HZCM              227.33429 Hz/(
```



Current Data Parameters

NAME	shawn_311a
EXPNO	2
PROCNO	1

F2 - Acquisition Parame

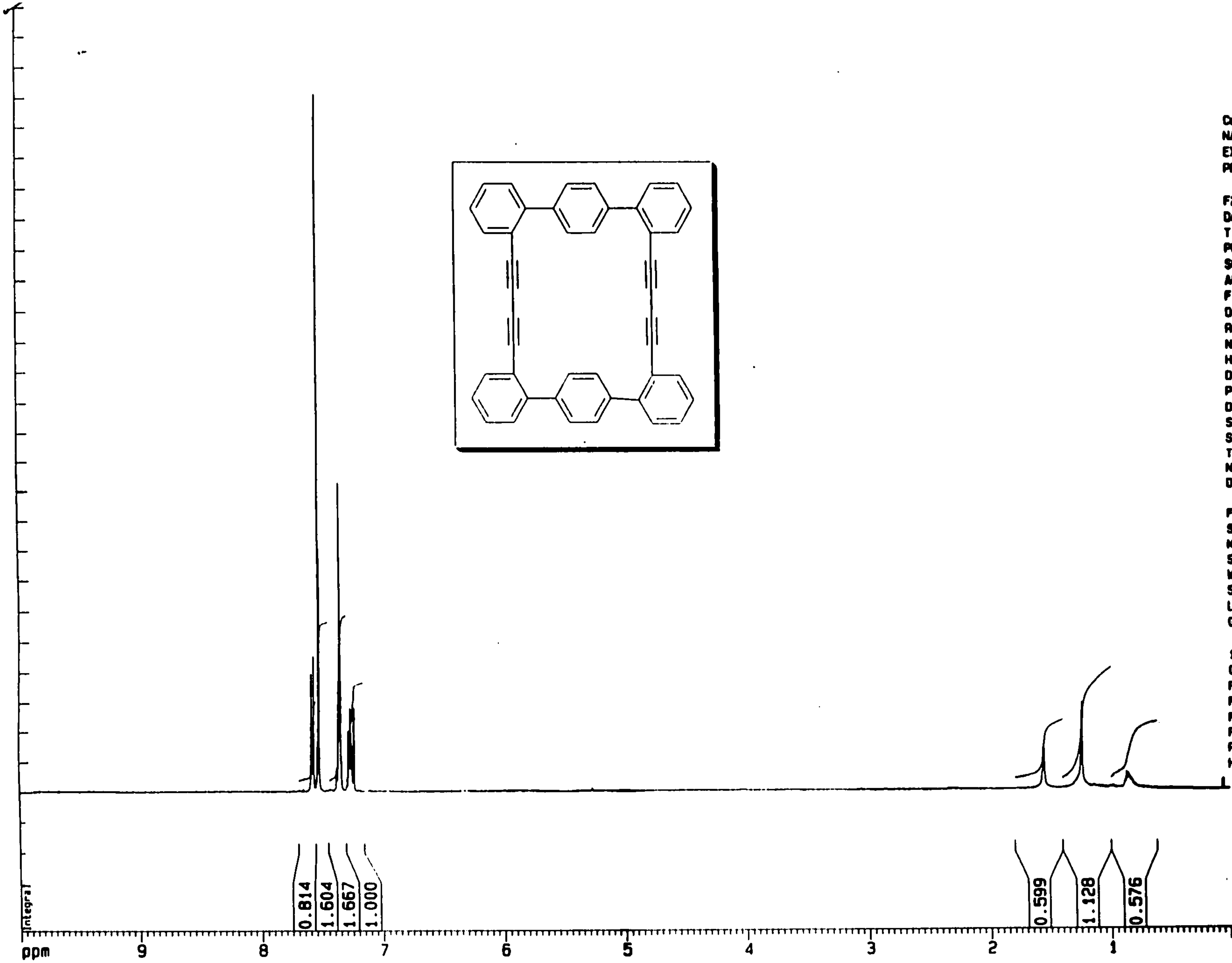
Date	990301
Time	13.21
PULPROG	zgdc
SOLVENT	CDCl3
AQ	1.0485760
FIDRES	0.476837
DW	16.0
RG	32768
NUCLEUS	13C
D11	0.0300000
P31	70.0
S2	22
HL1	22
D1	1.0000000
P1	5.0
DE	20.0
SFO1	125.7724464
SWH	31250.00
TD	65536
NS	1024
DS	0

F1 - Processing paramet

SI	32768
MC2	QF
SF	125.7591523
WDW	EM
SSB	0
LB	1.00
GB	0

1D NMR plot parameters

CX	22.00
F1P	200.000
F1	25151.83
F2P	0.000
F2	0.00
PPMCM	9.09091
HZCM	1143.26501

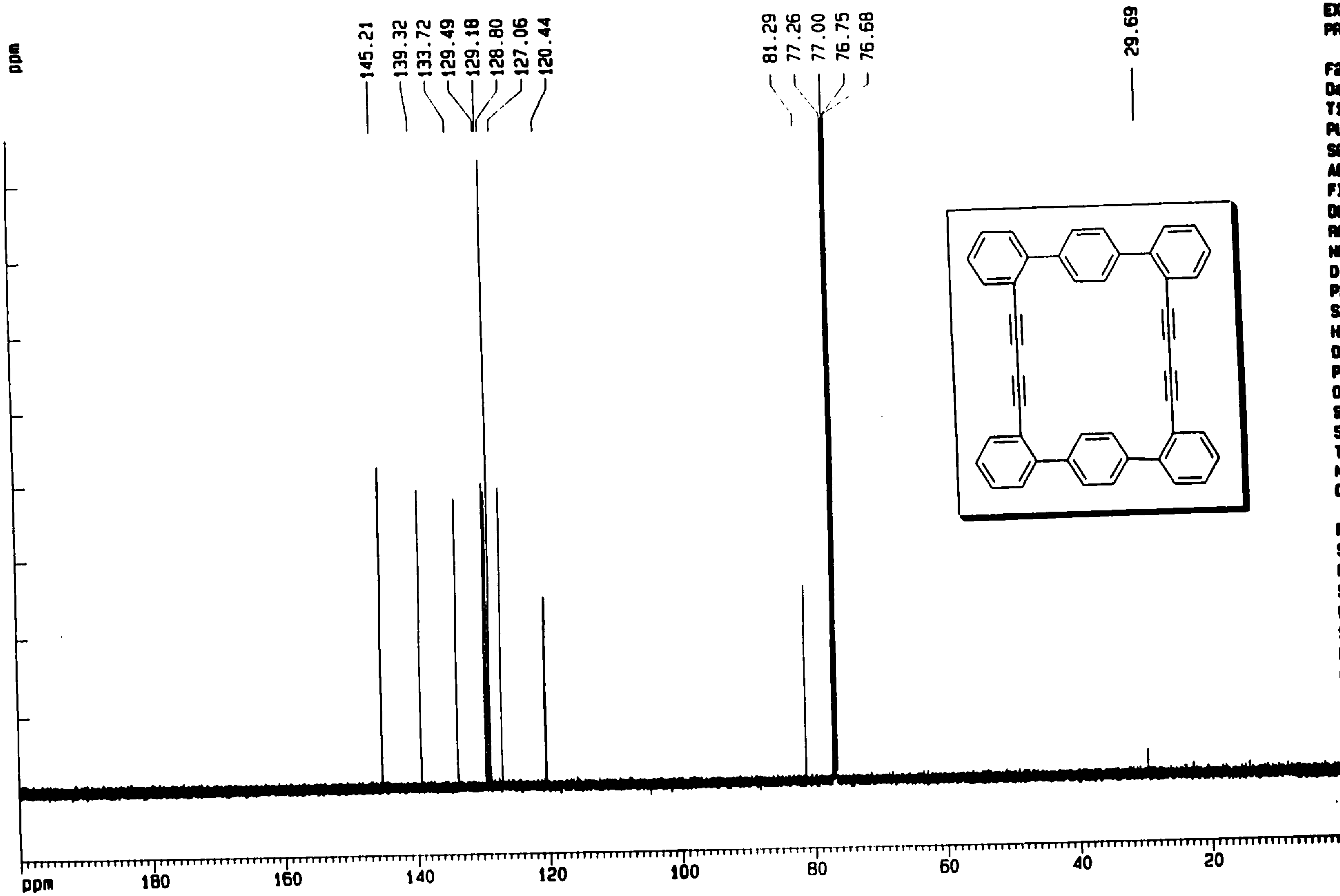


```
Current Data Parameters
NAME             sean_171b
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date                980525
Time                 16.23
PULPROG                 zg
SOLVENT              CDCl3
AQ            4.6530806 sec
FIDRES         0.107458 Hz
DW               71.0 usec
RG                    2048
NUCLEUS                 1H
HL1                   0 dB
D1          0.0100000 sec
P1                3.0 usec
DE               88.8 usec
SFO1       500.1381707 MHz
SWH            7042.25 Hz
TD                   65536
NS                      16
DS                       0

F1 - Processing parameters
SI                   32768
MC2                     QF
SF         500.1354311 MHz
WDW                     EM
SSB                      0
LB                 0.00 Hz
GB                       0

1D NMR plot parameters
CX                22.00 cm
F1P             10.000 ppm
F1             5001.35 Hz
F2P              0.000 ppm
F2                 0.00 Hz
PPMCM          0.45455 ppm/
HZCM         227.33429 Hz/c
```



Current Data Parameters

NAME	sean_171b
EXPNO	2
PROCNO	1

F2 - Acquisition Parame

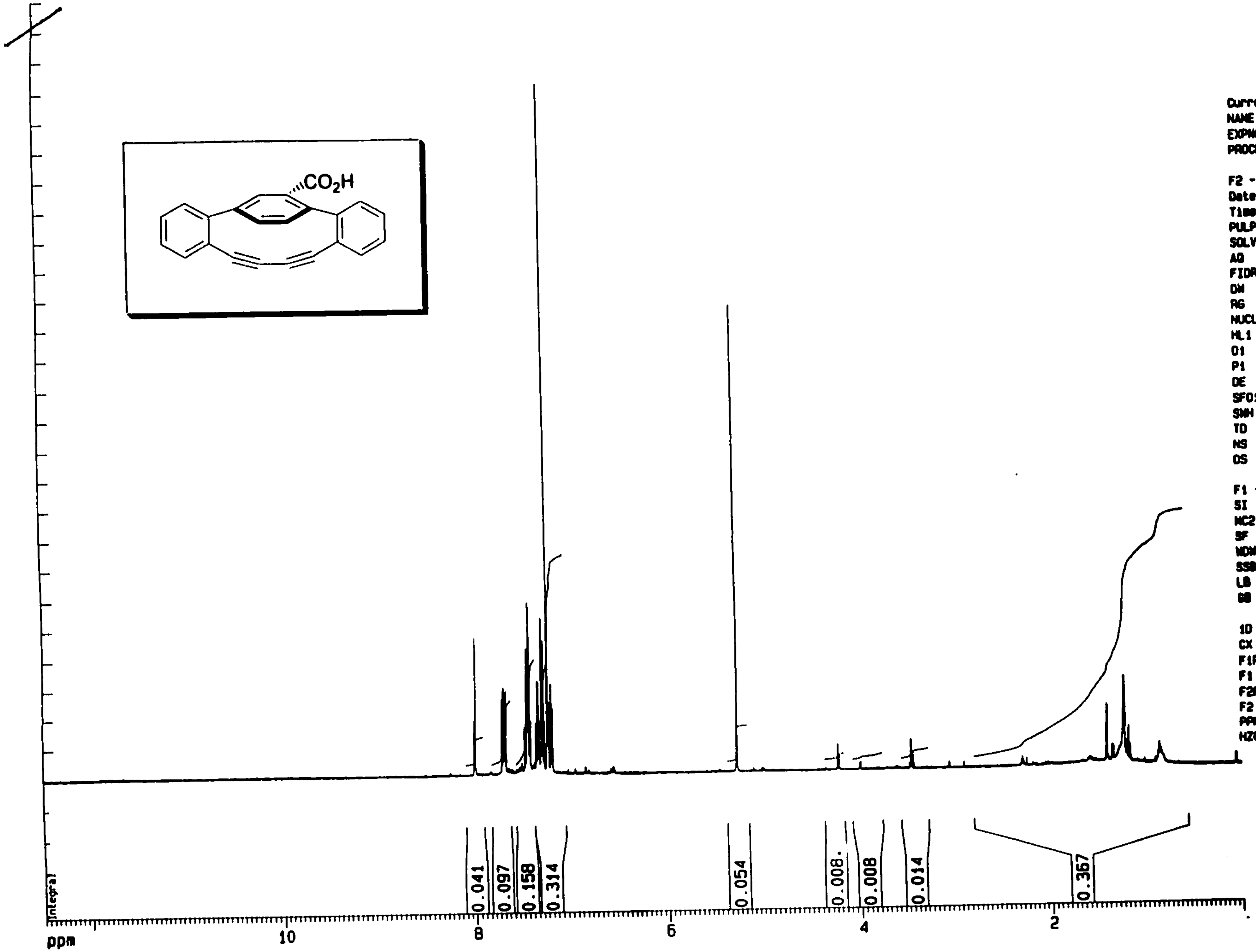
Date	980525
Time	16.55
PULPROG	zgdc
SOLVENT	CDCl3
AQ	2.9999881
FIDRES	0.166668
DW	16.0
RG	32768
NUCLEUS	13C
D11	0.0300000
P31	70.0
S2	22
HL1	22
D1	1.0000000
P1	5.0
DE	20.0
SFO1	125.7724464
SWH	31250.00
TD	187498
NS	13513
DS	64

F1 - Processing paramet

SI	131072
MC2	QF
SF	125.7591504
WDW	EM
SSB	0
LB	0.00
GB	0

1D NMR plot parameters

CX	22.00
F1P	200.000
F1	25151.83
F2P	0.000
F2	0.00
PPMCM	9.09091
HZCM	1143.26501

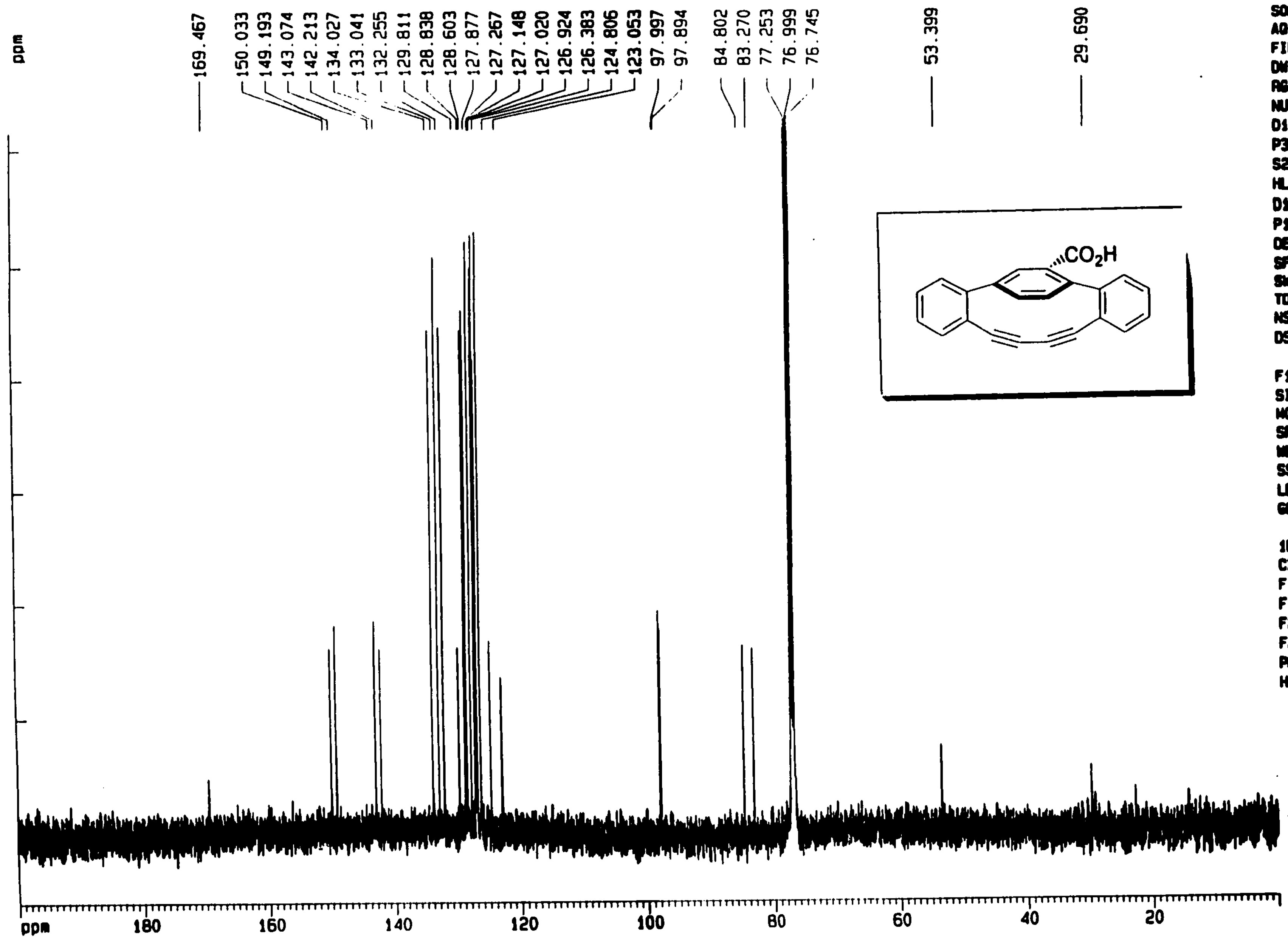


```
Current Data Parameters
NAME          shawn_330a
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date              990312
Time               16.26
PULPROG               zg
SOLVENT            CDCl3
AQ          4.6530806 sec
FIDRES       0.107456 Hz
DW              71.0 usec
RG                  4096
NUCLEUS               1H
HL1                 0 dB
D1          0.0100000 sec
P1               3.0 usec
DE              88.8 usec
SF01      500.1381707 MHz
SWH           7042.25 Hz
TD                 65536
NS                    32
DS                     0

F1 - Processing parameters
SI                 32768
MC2                   QF
SF        500.1354311 MHz
WDW                   EM
SSB                    0
LB               0.00 Hz
GB                     0

1D NMR plot parameters
CX              22.00 cm
F1P           12.500 ppm
F1            6251.89 Hz
F2P            0.000 ppm
F2               0.00 Hz
PPMCM        0.56818 ppm/
HZCM       284.16785 Hz/(
```

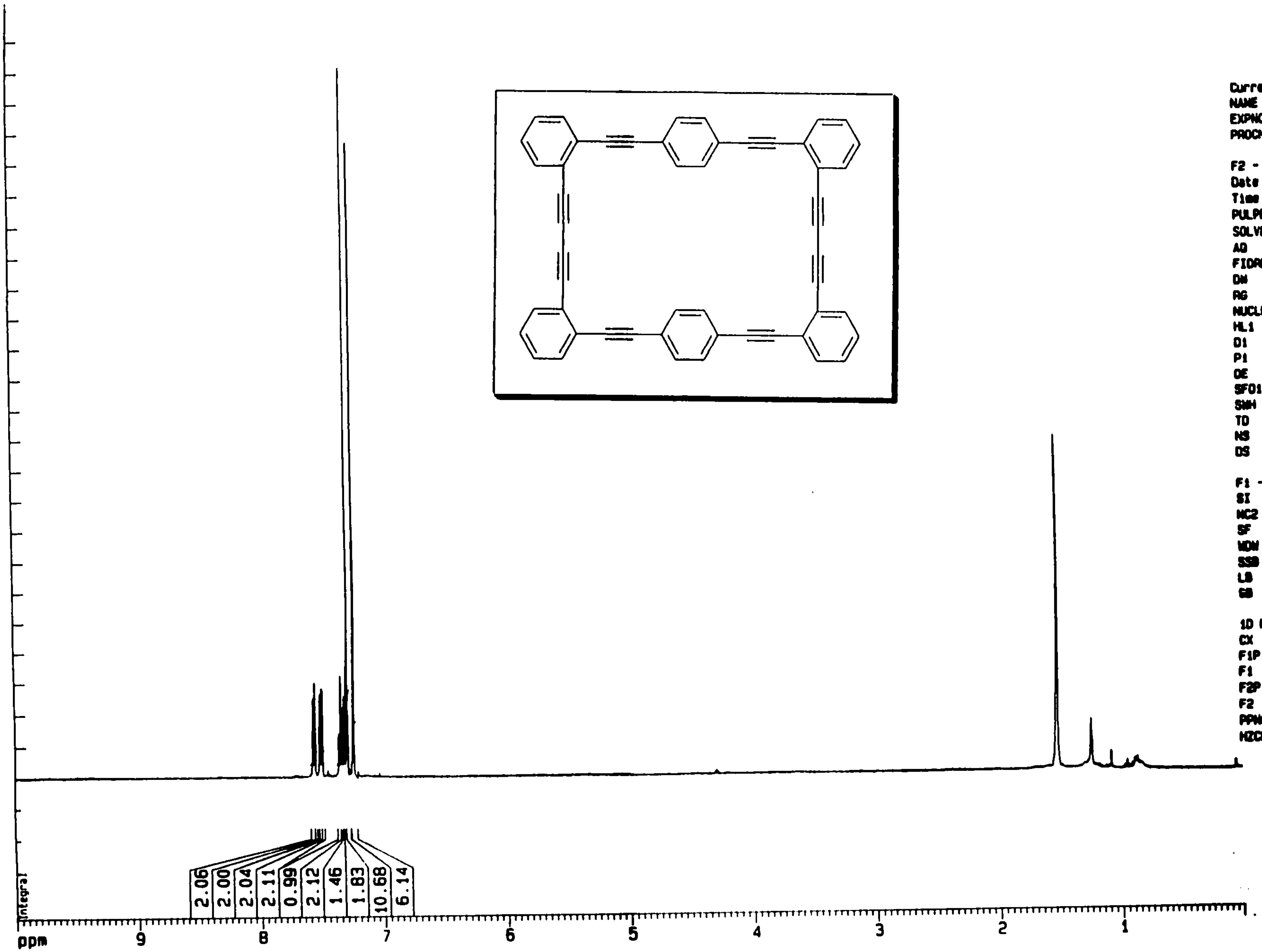


```
Current Data Parameters
NAME          shawn_339a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parame
Date              990312
Time               17.12
PULPROG             zgdc
SOLVENT            CDCl3
AQ             1.0485960
FIDRES          0.476837
DW                  16.0
RG                 32768
NUCLEUS              13C
D11           0.03000000
P31                 70.0
S2                    22
HL1                   22
D1            1.00000000
P1                   5.0
DE                  20.0
SFO1         125.7724464
SWH             31250.00
TD                 65536
NS                 32768
DS                     0

F1 - Processing paramet
SI                 32768
MC2                   QF
SF           125.7591495
WDW                   EM
SSB                    0
LB                  1.00
GB                     0

1D NMR plot parameters
CX                 22.00
F1P              200.000
F1              25151.83
F2P                0.000
F2                  0.00
PPMCM            9.09091
HZCM          1143.26501
```

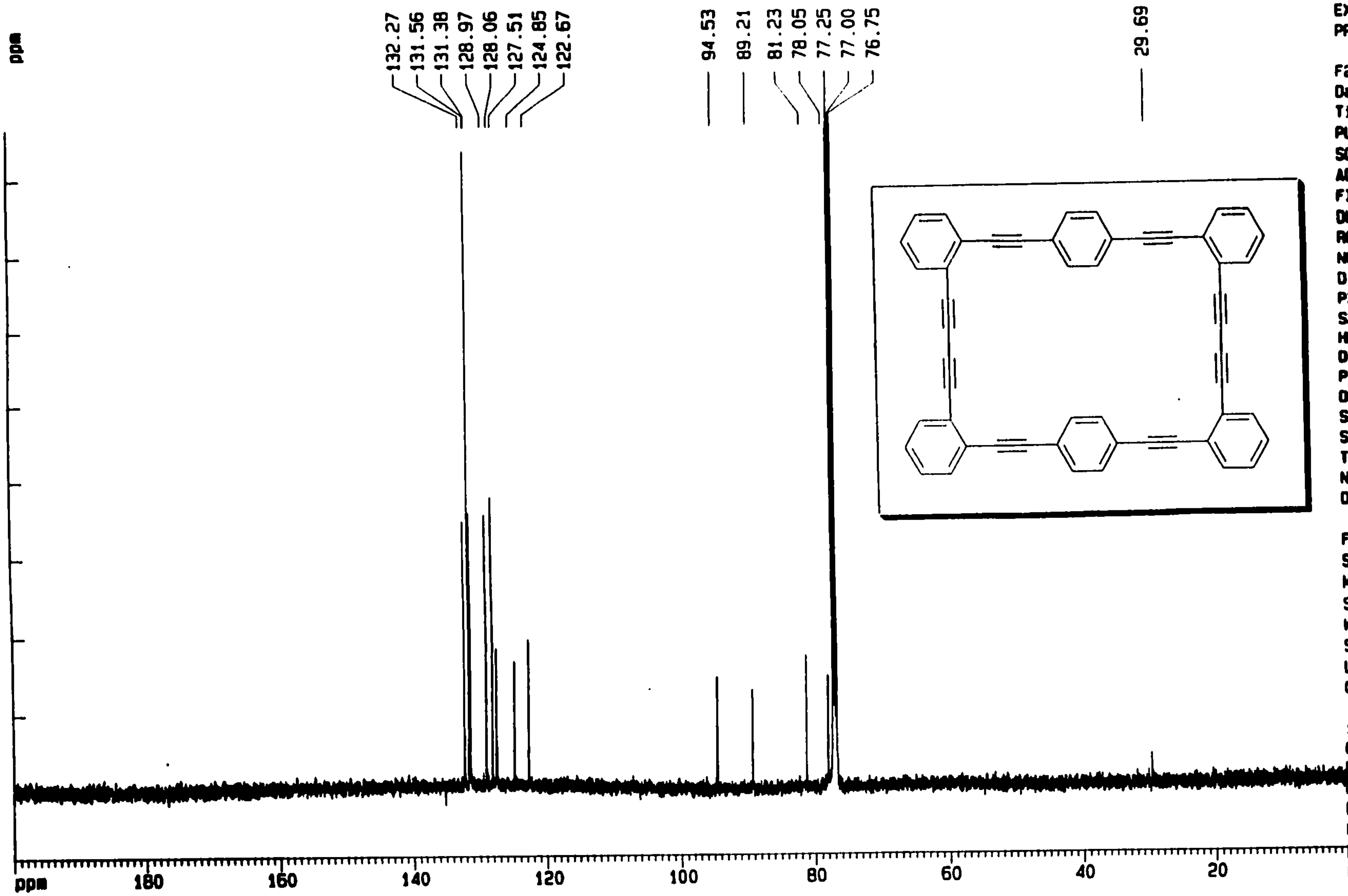


```
Current Data Parameters
NAME             shawn_257a
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date                 981016
Time                  17.28
PULPROG                  zg
SOLVENT               CDCl3
AQ             4.6530805 sec
FIDRES          0.107456 Hz
DW                 71.0 usec
RG                     4096
NUCLEUS                  1H
HL1                    0 dB
D1             0.0100000 sec
P1                  3.0 usec
DE                 88.8 usec
SFO1         500.1381707 MHz
SWH              7042.25 Hz
TD                    65536
NS                       64
DS                        0

F1 - Processing parameters
SI                    32768
MC2                      QF
SF           500.1354311 MHz
WDW                      EM
SSB                       0
LB                  0.00 Hz
GB                        0

1D NMR plot parameters
CX                  22.00 cm
F1P               10.000 ppm
F1                5001.35 Hz
F2P                0.000 ppm
F2                   0.00 Hz
PPMCM            0.45455 ppm/
HZCM           227.33429 Hz/(
```



```
Current Data Parameters
NAME          shawn_257a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parame'
Date              981016
Time               18.30
PULPROG             zgdc
SOLVENT            CDCl3
AQ             1.0485960
FIDRES          0.476837
DW                  16.0
RG                 32768
NUCLEUS              13C
D11            0.0300000
P31                 70.0
S2                    22
HL1                   22
D1             2.0000000
P1                   5.0
DE                  20.0
SFO1         125.7724464
SWH             31250.00
TD                 65536
NS                 51200
DS                     0

F1 - Processing paramet(
SI                 32768
MC2                   QF
SF           125.7591495
WDW                   EM
SSB                    0
LB                  1.00
GB                     0

1D NMR plot parameters
CX                 22.00
F1P              200.000
F1              25151.83
F2P                0.000
F2                  0.00
PPMCM            9.09091
HZCM          1143.26501
```

C(17)-C(18)-C(13)	120.45(17)
C(20)-C(19)-C(16)	176.82(19)
C(19)-C(20)-C(21)	175.68(19)
C(22)-C(21)-C(26)	119.18(15)
C(22)-C(21)-C(20)	121.25(15)
C(26)-C(21)-C(20)	119.52(15)
C(23)-C(22)-C(21)	120.99(16)
C(22)-C(23)-C(24)	119.65(16)
C(25)-C(24)-C(23)	120.87(16)
C(24)-C(25)-C(26)	119.99(16)
C(25)-C(26)-C(21)	119.23(14)
C(25)-C(26)-C(1)#1	120.39(15)
C(21)-C(26)-C(1)#1	120.35(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+3/2